QUALITY ASSURANCE PROJECT PLAN FOR THE CARBON REACTIVATION UNIT PERFORMANCE DEMONSTRATION TEST

Westates Carbon – Arizona, Inc.
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1.0 QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM AND DISTRIBUTION LIST

Project: Westates Carbon – Arizona, Inc. (WCAI)
RCRA Subpart X Performance Demonstration Test
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Key test Personnel Approvals and Distribution

Name/Function/Organization	Signature	Date
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QAPP Distribution List

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Sample Custodian	Sampling Contractor	1
Laboratory Analysis Coordinator	Contract Laboratory	1
Tribal Representative	Colorado River Indian Tribes (CRIT)	2
U.S. Environmental Protection Agency	EPA Region IX	2

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ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing Materials
B.P.	Boiling Point
CAR	Corrective action request
CCV	Continuing calibration verification
CEM	Continuous emissions monitor
CEMS	Continuous emissions monitoring system
CF	Calibration factor
CLP	Contract Laboratory Program
CMS	Continuous monitoring system
COC	Chain of Custody
CRIT	Colorado River Indian Tribes
CVAA or CVAAS	Cold vapor atomic adsorption spectroscopy
DI	Deionized (water)
DQO	Data quality objective
DRE	Destruction and removal efficiency
dscf	Dry standard cubic foot
dscfm	Dry standard cubic feet per minute
dscm	Dry standard cubic meter
dscmm	Dry standard cubic meters per minute
EDL	Estimated detection limit
EPA	U.S. Environmental Protection Agency
GC/MS	Gas chromatograph/mass spectrometry
g	grams
gr	Grains
HC	Hydrocarbons
HWC	Hazardous Waste Combustor
ICP or ICAP	Inductively coupled argon plasma spectroscopy
ICP-MS or ICAP-MS	Inductively coupled argon plasma spectroscopy/mass spectroscopy
ICV	Initial calibration verification
kg	Kilograms
L	Liter
LAC	Laboratory Analysis Coordinator
lb or lbs	Pounds

LCS	Laboratory control standard
MACT	Maximum Achievable Control Technology
MDL	Method detection limit
mg	Milligrams
μg or ug	Micrograms
MS	Matrix spike
MSD	Matrix spike duplicate
ND	Not Detected
ηg or ng	Nanograms
OCP	Organochlorine Pesticides
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PCDD	Polychlorinated dibenzo-p-dioxin
PCDF	Polychlorinated dibenzofuran
PDS	Post-digestion spike
PDT	Performance Demonstration Test
PDTP	Performance Demonstration Test Plan
PE	Performance evaluation
PIC	Product of incomplete combustion
POHC	Principal organic hazardous constituent
ppm	Parts per million
ppmv or ppmdv	Parts per million dry volume
QA	Quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality control
RCRA	Resource Conservation and Recovery Act
RDL	Reliable detection limit
RFA	Request for analysis
RPD	Relative percent difference
RSD	Relative Standard Deviation
RF	Response factor of Reactivation Furnace (note difference by context)
RSD	Relative standard deviation
RT	Retention time
SOP	Standard operating procedure
TCL	Target Compound List

THC	Total hydrocarbons
TOE	Total Organic Emissions
WCAI	Westates Carbon – Arizona, Inc.
WM	Wide-mouth
VOA	Volatile organic analysis
VOC	Volatile organic compound
VOST	Volatile organic sampling train

3.0 PROJECT DESCRIPTION

3.1 GENERAL

Westates Carbon – Arizona, Inc. (WCAI) has prepared a RCRA Part B permit application for it's Carbon Regeneration Furnace located in the Colorado River Indian Tribes (CRIT) Industrial Park near Parker, Arizona. WCAI has prepared the required RCRA Subpart X Performance Demonstration Test Plan (PDTP) which is designed to demonstrate the capability of RF unit to operate within the applicable emission limitations. WCAI has also been requested by EPA Region IX to perform a site-specific multiple pathway human health risk and ecological assessment as part of the permitting process. Accordingly, WCAI has prepared the PDTP and this Quality Assurance Project Plan (QAPP) to incorporate the gathering of emissions data to demonstrate compliance with the applicable regulatory requirements and for use in the risk assessments. Specific guidance issued by EPA (Risk Burn Guidance for Hazardous Waste Combustion Facilities, EPA530-R-01-001, July 2001) has been used, along with WCAI's Risk Assessment Protocol document, to identify compounds of potential concern for the risk assessment and to select appropriate sampling and analytical techniques.

3.2 TEST SCOPE

The WCAI Performance Demonstration Test Plan has been prepared to provide comprehensive performance testing of the RF unit to demonstrate compliance with the applicable HWC MACT emission standards and to gather data for use in a site-specific risk assessment. The objectives of the PDTP are to demonstrate regulatory compliance with standards such as Destruction and Removal Efficiency (DRE) and particulate matter emission concentration, while operating at "worst case" conditions processing normal feed materials, which have been augmented with metals, chloride, etc., to establish operating conditions that will be included in the permit. More specifically, the objectives of the Performance Demonstration Test (PDT) are as follows:

3.2.1 Test Objectives

1. Demonstrate Compliance with Applicable USEPA Regulatory Performance Standards (Based on HWC MACT Standards for Existing Hazardous Waste Incinerators):

- Demonstrate a DRE of greater than or equal to 99.99% for the selected principal organic hazardous constituents (POHCs) chlorobenzene and tetrachloroethene.
- Demonstrate stack gas carbon monoxide concentration less than or equal to 100 ppmv, dry basis, corrected to 7% oxygen.
- Demonstrate stack gas hydrocarbon concentration of less than or equal to 10 ppmv, as propane, dry basis, corrected to 7% oxygen.
- Demonstrate a stack gas particulate concentration less than or equal to 34 mg/dscm (0.015gr/dscf) corrected to 7% oxygen.

- Demonstrate that the stack gas concentration of hydrogen chloride (HCl) and chlorine (Cl2) are no greater than 77 ppmv, dry basis, corrected to 7% oxygen, expressed as HCl equivalents.
- Demonstrate that the stack gas mercury concentration is less than or equal to 130 μg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of semivolatile metals (cadmium and lead, combined) is less than or equal to 240 μg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of low volatility metals (arsenic, beryllium, and chromium, combined) is less than or equal to 97 μg/dscm, corrected to 7% oxygen.
- Demonstrate that the stack gas concentration of dioxins and furans does not exceed 0.40 ng/dscm, corrected to 7% oxygen, expressed as toxic equivalents of 2,3,7,8-TCDD (TEQ). This is the applicable standard since the gas temperature entering the first particulate matter control device is less than 400°F.

2. Establish Permit Operating Limits

- Demonstrate maximum feed rate for spent activate carbon.
- Demonstrate minimum afterburner gas temperature
- Demonstrate maximum combustion gas velocity (or a suitable surrogate indicator)
- Demonstrate maximum total chlorine/chloride feed rate
- Establish a Maximum Theoretical Emission Concentration (MTEC) limit for mercury
- Demonstrate system removal efficiency (SRE) for semivolatile and low volatility metals so feed rate limits can be developed by extrapolation from test results.
- Establish appropriate operating limits for the air pollution control system components.

3. Gather Information for Use in a Site-Specific Risk Assessment

- Measure emissions of metals, including hexavalent chromium
- Measure emissions of specific volatile and semivolatile products of incomplete combustion (PICs)
- Measure emissions of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF)
- Measure emissions of polychlorinated biphenyls (PCBs)
- Measure emissions of specific organochlorine pesticides
- Measure emissions of total volatile, semivolatile, and nonvolatile organics
- Determine the stack gas particle size distribution.

3.2.2 Test Protocol Summary

In order to accomplish the PDT objectives, (i.e., demonstrating that the unit will meet all applicable environmental performance standards) a single test condition representing “worst case” operations of minimum temperature, maximum combustion gas velocity (minimum residence time), and maximum spent activated carbon feed rate will be performed. The test will consist of at least three replicate sampling runs.

A summary description of the testing conditions, analytical parameters, and sampling methods follows:

3.2.3 Test Condition 1 ("Worst-Case" Operations)

Sampling and monitoring protocols that will be utilized while carrying out the performance test are summarized as follows:

- Spent Activated Carbon Feed - total chlorine/chloride, elemental (C, H, N, O, S, moisture), volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Makeup Water - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Caustic feed to APC - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Cu, Co, Pb, Hg, Ag, Tl, Se, Ni, V, Zn)
- Scrubber Blowdown - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Wastewater Discharge to POTW - volatile organics, semivolatile organics, and total metals (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, Zn)
- Stack gas particulate, HCl, and Cl_2 using EPA Method 26A
- Stack gas target volatile organics using VOST, SW-846 Method 0030
- Stack gas target semivolatile organics and organochlorine pesticides using SW-846 Method 0010
- Stack gas PAHs and PCBs using a separate SW-846 Method 0010 sampling train
- Stack gas PCDD/PCDF using SW-846 Method 0023A
- Stack gas total volatile organics using SW-846 Method 0040
- Stack gas total semivolatile and nonvolatile organics using SW-846 Method 0010
- Stack gas metals (Al, Sb, As, Ba, Be, Cd, total Cr, Co, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn) using EPA Method 29
- Stack gas hexavalent chromium using SW-846 Method 0061
- Stack gas particle size distribution using a EPA Method 5 sampling train with smooth filter media
- Stack gas CO and O_2 by permanently installed CEM according to the protocols in the Appendix to 40 CFR 63, Subpart EEE; Performance Specification 4B of 40 CFR 60, Appendix B.
- Stack gas total hydrocarbons (as propane) by temporary CEM according to EPA Method 25A and the protocols in the Appendix to 40 CFR 63, Subpart EEE.

3.3 QUALITY ASSURANCE PROJECT PLAN SCOPE

This QAPP presents the organization, objectives, functional activities and specific Quality Assurance (QA) and Quality Control (QC) activities for the Performance Demonstration Test to be performed at the WCAI Carbon Reactivation facility near Parker, Arizona. This QAPP also describes the specific QA/QC protocols that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory analysis during the test program. The QAPP is an integral part of the PDT plan and must be used in conjunction with the PDT plan.

All QA/QC procedures will be in accordance with applicable professional technical standards, government regulations and guidelines, and specific project goals and requirements. This QAPP has been prepared in accordance with EPA QAPP guidance documents, in particular the following:

1. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80)
2. Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January 1990.

4.0 ORGANIZATION OF PERSONNEL, RESPONSIBILITIES, AND QUALIFICATIONS

The project organization for this test is summarized in Figure 4-1.

All test activities are conducted under the overall direction of the WCAI Plant Manager, and in accordance with WCAI policies administered by the Director of Environmental Health and Safety. During the test, the WCAI Plant Manager will be responsible for ensuring that the process runs properly and that the unit achieves the desired test conditions on each test day. As such he will assign responsibilities to the unit operators. This individual will also be responsible for ensuring that all of the applicable process data are collected during each of the test runs. The WCAI Director of Plant Operations will be responsible for supervising all of the contractors associated with the program and will serve as the official communication link between WCAI and the respective contractors and regulatory observers. The contractors include the Test Coordinator, Sample Custodian, Feed Spiking Operator, Process Sampling Coordinator, Stack Sampling Coordinator, and the Laboratory Analysis Coordinator.

The WCAI Director of Environmental Health and Safety is responsible for environmental compliance activities related to the plant site. During the testing, the WCAI Director of Environmental Health and Safety and other environmental staff members will be available to lend support to the testing program where needed.

The project team consisting of the WCAI Director of Plant Operations and representatives of the contractors will implement the test programs. The Test Coordinator will be a consulting contractor who is experienced in the technical coordination and QA/QC associated with the testing of hazardous waste thermal treatment systems. The stack sampling for this project will be conducted by a contractor who is experienced in conducting the stack sampling called for in the PDT plan. Analytical services will be provided by a contract laboratory(s) experienced in the analysis of stack emissions test samples and waste samples.

The Test Coordinator is responsible for the execution of the PDT plan, the QAPP, the preparation of the Test Reports, and the interpretation of the results of the tests. During the tests, the Test Coordinator is responsible for the overall implementation of the tests. The Test Coordinator will serve as the focal point between the WCAI Plant Manager, WCAI Director of Plant Operations, and the sampling contractors on testing related matters, and will coordinate activities among various project team members. Specific responsibilities will include:

- Ensuring compliance with the PDT plan and the QAPP by all project team members during the test

- Documenting testing activities in a field logbook
- Assisting WCAI in interfacing with the regulatory observers and/or oversight contractors during the test
- Providing coordination among WCAI personnel and the sampling team during the test, especially regarding decisions to start, stop, hold or repeat sampling runs
- Providing field review of process operating logs, sample collection sheets, stack sampling logs, chain of custody forms, and request for analysis forms
- Interfacing with the Laboratory Analysis Coordinator while samples are being analyzed
- Interfacing with the Stack and Process Sampling Coordinators while the sampling data is being reduced
- Supervising production of the Test Report
- Certifying the overall PDT Results and PDT Report
- Preparing operating specifications for the systems based on the results of the test.

A Quality Assurance Officer (QAO) will be appointed whose responsibilities will include the following:

- Reviewing QA/QC activities and communicating the results of those activities to the appropriate personnel (refer to Figure 4-1)
- Making recommendations to WCAI and the Test Coordinator regarding any problems that may be detected
- Ensuring that the sample preservation and shipments are being properly monitored by the Sample Custodian and that any samples with preservation or holding time exceedances are reported to the Test Coordinator and WCAI immediately
- Ensuring that appropriate corrective actions are taken if problems are detected
- Conducting or coordinating any required audits of field, office, or laboratory procedures to ensure compliance with the QAPP
- Verifying that test data are adequately recorded and maintained and that data are properly reduced, validated, and interpreted
- Assuring all individuals included in the QAPP Distribution List receive current copies of revisions as applicable
- Performing inspections of the process equipment, process controls, process operations, data acquisition and recording systems, and sampling activities for compliance with this QAPP and the PDT plan
- Performing audits of the laboratories for compliance with this QAPP and the PDT plan
- Reviewing the stack sampling and analytical reports for completeness and accuracy
- The results of the above mentioned inspections will be documented in a written report, included in the PDT report.

A Process Sampling Coordinator will be appointed who will have overall responsibility for the collection and handling of all process related samples other than stack gas related samples. The Process Sampling Coordinator may also be the Sample Custodian and has the following responsibilities:

- Coordinating the preparation and shipment of process sampling equipment, and shipping containers to the test site
- Directing and/or participating in sampling activities
- Recording field test data required by the PDT plan or sampling methods
- Reviewing and approving sample collection sheets and field data sheets
- Documenting process sampling activities in a field logbook
- Overseeing recovery of samples and preservation of samples in the field
- Performing all QA activities required by the sampling methods
- Preparing a draft and final report of process sampling activities.
- Documenting all samples taken.

A Stack Sampling Coordinator will be appointed who will have overall responsibility for the collection and handling of all stack samples. The Stack Sampling Coordinator has the following responsibilities:

- Preparing and shipping stack sampling equipment, and shipping containers to the test site
- Preparing and calibrating stack sampling equipment
- Directing and/or participating in stack sampling activities
- Recording field test data required by the PDT plan and stack sampling methods
- Reviewing and approving stack sample collection sheets and stack sampling field data sheets
- Documenting stack sampling activities in a field logbook
- Overseeing recovery of stack samples and preservation of those samples
- Reducing stack sampling data and performing all calculations and QA activities required by the stack sampling methods
- Preparing a draft and final report of stack sampling activities.
- Notify the Sample Custodian of all samples taken.

The WCAI Process Operators will be responsible for the operation of the RF. Their duties will include:

- Maintaining the RF systems within specified target limits
- Maintaining logs of process data as required
- Downloading and providing on 3.5-inch or compact disc in Microsoft Excel or ASCII format the RF one-minute operating data to the Test Coordinator
- Collecting selected process samples
- Notifying the Sample Custodian of all samples taken.

A Sample Custodian will be responsible for handling all samples collected during the test. His/her duties will include:

- Assigning and recording sample numbers

- Preparing samples and packaging them for shipment to the laboratory
- Preparing chain of custody (COC) and request for analysis (RFA) forms for all samples
- Coordinate shipping of all samples to the laboratory.
- Monitoring the shipment of samples to the laboratory to ensure that all samples are received on schedule and with all preservation requirements being met (Any discrepancies should be immediately reported to the QAO, Test Coordinator and WCAI)

A Laboratory Analysis Coordinator (LAC) will be appointed for the laboratory that provides analytical services for the project. His/her responsibilities will include:

- Receiving, verifying, and documenting that incoming field samples correspond to the sample chain of custody information
- Notifying the Sample Custodian, QAO, Test Coordinator and WCAI of any discrepancies or problems in the chain of custody information, preservation, or sample condition
- Maintaining records of incoming samples
- Tracking samples through processing, analysis, and disposal
- Preparing QC samples for analysis during the project
- Verifying that personnel are trained and qualified in specified laboratory QC and analytical procedures
- Verifying that laboratory QC and analytical procedures are being followed as specified in the this QAPP and the laboratory specific QA/QC Plan and analytical standard operating procedures (SOPs)
- Reviewing QC and sample data during analysis and determining if repeat samples or analyses are needed
- Submitting certified QC and sample analysis results and data packages to the Test Coordinator
- Archiving analytical data.

Resumes of key individuals who will be implementing the test are presented in Appendix A.

5.0 QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL OBJECTIVES

5.1 GENERAL

The overall quality assurance objective of this test project is identifying the complete set of data necessary to provide a complete quality assessment of the test results. These data include all the quality indicators generated during the project, and the adherence of the test data to the acceptance criteria for precision and accuracy that are used to assess the data quality. The specific quality objective is to produce a complete data set that can be used to fully assess and validate the RF's operation relative to the applicable emissions and performance standards.

The field and laboratory data obtained during this test will be reviewed by the Quality Assurance Officer, and a complete assessment of the data quality indicators will be included in the final test report. The data quality will be discussed with respect to meeting the respective data quality objectives (DQOs) and the overall project objective. Data that is outside of the target DQO limits will be evaluated relative to the impact(s) on the overall project objective of assessing the RF system's performance. This data evaluation and validation will be included in the test report.

Table 5-1 presents target DQOs for precision and accuracy for each type of analysis that will be performed during the test program. QA/QC objectives for precision, accuracy, representativeness, completeness, comparability, and sensitivity are defined in this section. Procedures and formulas for determining accuracy and precision are presented in Section 13.0 of this document. The following definitions briefly describe the meaning of each QA/QC objective:

Precision: A measure of mutual agreement among individual measurements of the same property, usually under "prescribed similar conditions." Various measures of precision exist depending on the prescribed similar conditions. If the number of samples is less than three, the precision is described as range percent or relative percent difference (RPD) from the average of replicate measured values for analysis of the same parameter. If the number of samples is three or greater, precision is best described in terms of relative standard deviation (RSD).

Accuracy: The degree of agreement of a measurement (or an average of measurements of the same parameter) X, with an accepted reference or true value, T. Accuracy is usually expressed as the difference between the two values, X - T, or the difference as a percentage of the reference or true value, 100 (X - T)/T, and sometimes expressed as a ratio, X/T. In some cases, accuracy is described as the percentage recovery of a known quantity of material added to a sample prior to analysis. Accuracy is a measure of the bias in a system.

Completeness: A measure of the amount of valid data obtained compared to the amount expected to be collected under normal conditions. Completeness is usually expressed as a percentage.

Representativeness: The degree to which data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, process condition, or an environmental condition.

Comparability: The confidence with which one set of data can be compared to another.

Sensitivity: The ability of a measurement system to accurately and precisely determine a desired property within the limits needed to assess the measurement result against established criteria. For this type of program the required sensitivity is a function of assessment criteria, sample size, and analytical detection limit.

5.2 PRECISION AND ACCURACY

A number of procedures will be used to meet the precision and accuracy objectives of the analytical program. All sampling and analytical activities will be conducted following referenced procedures. All reference materials used as calibration standards, surrogate compounds, or laboratory control samples will be of the highest purity commercially available. Contract Laboratory Program (CLP) compounds for matrix spikes and surrogates will be used for organic analyses by gas chromatograph/mass spectrometry (GC/MS). All spiking levels will be in accordance with the referenced methods. Table 5-2 lists the organic compounds and the applicable control limits of laboratory surrogates and field spikes to be used to spike samples. The calibration of instruments used during analysis will be verified each day that samples are analyzed as described in later sections of this QAPP. Assessment of data precision and accuracy will be accomplished by evaluating the results from multiple analyses of the same parameter, and analysis of standards, duplicates and spiked samples. Field and laboratory contamination will be assessed through the analysis of reagent, instrument, method, field and trip blanks.

Precision estimates presented in Table 5-1 represent variability for replicate measurements of the same parameters, expressed in terms of relative percent difference (RPD) for duplicate samples or relative standard deviation (RSD) for three or more measurements, as appropriate. For analyses of samples with detectable concentrations of the target analytes, precision is evaluated by conducting duplicate analyses of unspiked samples and assessing the RPD. In the evaluation of larger data sets (three or more data points), the RSD is assessed. When duplicate analyses are performed, the average of the original and duplicate results is used in test calculations. If the variance in the duplicate analyses bring into question the analytical precision, additional analyses, if allowed by the method, will be performed to better determine the actual value or to evaluate the potential reason(s) for the measurement variability.

For analytical results near the detection limit, precision can be impacted. For the cases where the original and duplicate results are a combination of detect and non-detect results at the method detection limit (MDL) where precision can not be calculated, the data will be flagged as estimated.

Accuracy values in Table 5-1 include components of both random error and bias, expressed as a percentage of the “true” of “known” value (for reference materials) or percent analyte recovery (for spiked samples). The QA/QC program will focus upon controlling measurement error within the estimated limits of measurement uncertainty, as specified in Table 5-1. It should be noted that these limits are estimates that are, in most cases, described in the referenced analytical methods or in QA/QC guidance for hazardous waste incineration. They represent the range of results that can be expected from these methods based on actual field sampling results and laboratory-based QA/QC studies. Therefore, it is reasonable to expect that the measurement errors associated with this project will be within the objectives shown in Table 5-1. QA/QC determinations which fall outside of the target range will be flagged and an assessment of the impact, if any, on the usefulness of the data and the overall results and conclusions of the test program will be provided in the Test Report. Specifically, if Matrix Spike/Matrix Spike Duplicate (MS/MSD) percent recoveries fall outside the control limits the Laboratory Control Samples (LCSs) and field blanks will be reviewed to determine the effect of the matrix on spike recovery.

If ongoing QA/QC procedures reveal that a measurement's error has exceeded the estimated data quality limits, the source of the excessive error will be identified and corrective action will be taken, as described in Section 14.0. If data fall outside the acceptable range of precision and accuracy, even after corrective action has been taken, those data points will be flagged in the final report. The precision and accuracy for those measurements will be reported as determined using the actual data. Also, alternative procedures (either sampling or analytical) may be considered and recommended if possible.

The analytical laboratory conducting the analysis of the samples will be required to have standard operating procedures (SOPs) for each analysis to be performed. The laboratory will also be required to have procedures for preparing, reviewing, modifying, and controlling distribution of analytical procedures.

5.2.1 CEMS Precision and Accuracy

The precision of the installed carbon monoxide and oxygen continuous emissions monitoring system (CEMS) analyzers will be assessed during the test using the recommended calibration gases in accordance with 40 CFR 60, Appendix B, Specification 4B. The precision of the temporary hydrocarbon analyzer to demonstrate compliance with 40 CFR 63.1203(b)(5)(ii), as allowed by 40 CFR 63.1206(b)(6) will be assessed during the test using the recommended calibration gases in accordance with 40 CFR 60, Appendix A, Method 25A. Precision will be assessed using the following equation:

$$\text{Precision}(\%drift) = \left(\frac{R_f - R_i}{Span}\right)x100$$

where:

R_f = Final monitor response at end of the test run

R_i = Initial monitor response at start of the test run

Span = Maximum range of the analyzer.

The accuracy of all CEMS analyzers will be evaluated during the test by the measurement of percent accuracy as defined by the equation below:

$$\text{Accuracy}(\%) = \left(\frac{R_a - R_c}{Span}\right)x100$$

where:

R_a = Analyzer indicated concentration of the calibration gas

R_c = Certified concentration of the calibration gas

Span = Maximum range of the analyzer.

The accuracy of oxygen and carbon monoxide CEMS analyzers will be evaluated in accordance with the CMS/CEMS Performance Evaluation Test Plan conducted prior to the test. This test will include calibration drift tests, response time tests, calibration error tests, and relative accuracy tests per the 40 CFR 60, Appendix B, Specification 4B.

5.2.2 VOST Precision and Accuracy

The Volatile Organic Sampling Train (SW-846, Method 0030) will be used to sample stack gases for the volatile POHCs and volatile PICs during the PDT. Prior to their use in the field, two pairs of the Tenax and Tenax/charcoal tubes from the batch of tubes prepared for specifically this project will be spiked with the project-specific volatile surrogates and matrix spike compounds and analyzed prior to field sample analysis. The precision assessment for VOST requires that the RPD associated with each analyte be $\leq$25% for these spiked resin blanks. Additional precision data for the actual samples are obtained by calculating the RSD associated with surrogate spikes applied to each VOST sample. The variation of surrogate recoveries should be $\leq$35% RSD for actual VOST analyses.

VOST accuracy is best assessed via a blind audit. The VOST audit includes VOST sampling of a compressed gas cylinder containing a mixture of volatile organic compounds in nitrogen. During the VOST audit, the VOST is operated by the same technician that will perform the stack sampling. The VOST audit kit, which includes the audit cylinder, cylinder heater and connecting adapters, is provided by the regulatory observers. Using the same VOST apparatus and VOST tubes prepared for the test, the technician will setup the audit cylinder per the accompanying directions and sample its contents. A minimum of four tube set pairs will be collected. The VOST audit samples are than submitted for analysis along with the other test VOST samples. The concentrations of the volatile organics in the cylinder determined via the VOST audit sample analyses are included in the QA/QC section of the test report. Only the concentrations of volatile compounds from the target list of volatile compounds for this test program will be included in the reported audit results.

The VOST Audit Cylinder Gas Coordinator is an independent government office located in Research Triangle Park, North Carolina. The identity of the compounds and concentrations of the compounds in the VOST audit cylinder are not known to the regulatory observers. The VOST audit kit may only be requested by the observing regulatory agency, and must be provided to the test site by the regulatory observers for sampling by the auditee. The VOST kit must remain in the custody of the regulatory observers at all times. The VOST audit kit and cylinder seals must remain intact until they are broken by the regulatory observers at the time of the on-site audit. The VOST audit procedure must be continually observed by the regulatory observers. The auditee may only sample the audit cylinder using the VOST; NO OTHER SAMPLING OF THE CYLINDER SUCH AS BAG SAMPLES IS ALLOWED. At the end of the audit, the VOST kit must be secured and retained by the regulatory observers for return to the Audit Cylinder Gas Coordinator.

The regulatory observers must forward the VOST audit results from the test report to the Audit Cylinder Gas Coordinator for review. The VOST audit is a simply pass/fail audit. The successful VOST audit will determine the concentrations of specific volatile organic compounds within 50-150% of the known value, known only by the Audit Cylinder Gas Coordinator. The Audit Cylinder Gas Coordinator will issue a pass/fail determination to the regulatory observers.

Recent VOST audit experience has shown that the VOST audit procedures must be modified slightly from the directions provided with the VOST audit kit and the way stack gas is actually sampled. Specifically, the standard sample volume per tube set is ~20 liters of stack gas. However, if similar size samples were collected from the VOST audit cylinder, the concentrations of organic analytes can saturate the tubes, and may exceed the range of the Method 5041/6260 analysis (typically >1000 ηg) and may not meet the criterion noted in the preceding paragraph. VOST audit experience has shown that this situation can usually be precluded if the VOST audit sample volumes are limited to 2.5 to 5 liters per tube set.

A more qualitative evaluation of accuracy for the VOST is prescribed in Section 7.3.7 of the Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January 1990. This reference requires that the Tenax and Tenax/charcoal VOST tubes from each set of test run tube pairs be analyzed separately to determine possible POHC breakthrough to the Tenax/charcoal tube. The analysis of a Tenax/charcoal tube should indicate less than 30% of the POHC concentration that is collected on the Tenax tube. Breakthrough of the POHC to the Tenax/charcoal tube above this level may cause loss of the desorption efficiency and result in a low bias in the analytical result. This criterion does not apply when less than 75 ηg of POHC is detected on the Tenax/charcoal tube.

5.2.3 Method 0010 Semivolatile Organic Sampling Precision and Accuracy

A SW-846 Method 0010 sampling train, sometimes referred to as the Modified Method 5 (MM5) sampling train, will be used to sample the stack gas for semivolatile organic compound (SVOC) PICs including PAHs, OC Pesticides, and PCBs. Prior to use in the field, the XAD-2 resin traps for use in the Method 0010 sampling train are spiked with isotopically labeled versions of semivolatile organics noted in Table 5-2. Prior to their use in the field, two of the prepared XAD-2 resin traps from the batch of traps prepared specifically for this project will be analyzed prior to field sample analysis. The precision assessment for Method 0010 sampling train requires that the RPD associated with each semivolatile analyte be $\leq$50% for the spiked semivolatile, PAH, OC Pesticides, and PCB compounds. The variation of surrogate recoveries should be $\leq$40% RSD for actual Method 0010 analyses.

5.2.4 Method 0023A Dioxin/Furan Sampling Precision and Accuracy

A SW-846 Method 0023A sampling train will be used to sample the stack gas for polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/PCDFs). Prior to use in the field, the XAD-2 resin traps for use in the Method 0023A sampling train are spiked with isotopically labeled versions of the PCDD/PCDFs noted in Table 5-2. Prior to their use in the field, two of the prepared XAD-2 resin traps from the batch of traps prepared specifically for this project will be analyzed prior to field sample analysis. The precision assessment for Method 0023A sampling train requires that the RPD associated with each analyte be $\leq$30% for the spiked compounds. The variation of surrogate recoveries should be $\leq$30% RSD for actual Method 0023A analyses.

5.3 DETECTION LIMITS AND REPORTING

How the detection limits will be used in data reduction and reporting is also described in Section 11.3.3. For inorganic analyses and non-isotope dilution method organic analyses, the laboratory report will provide both the sample specific Method Detection Limit (MDL) and the Reliable Detection Limit (RDL). The laboratory will also provide a description detailing how each detection limit and reporting limit was derived. All non-detects for target analytes will be reported at the laboratory-determined MDL. If the analyte is detectable at some value between the MDL and RDL, the detected value will be reported and flagged as estimated. For isotope dilution organic analysis methods, the non-detects for the isotope dilution methods will be determined using the method specific SW-846 definition of an estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory. The verification of the analytical methods and MDLs for non-detect values relative the project specific matrices is discussed in the following sections. Estimates stack gas detection limits for this project are presented in Table 5-3.

5.3.1 Detection Limit Definitions

MDL – method detection limit – The minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero. It is a statistical limit that is matrix dependent. The MDL is generally derived following SW-846 Chapter 1 Section 5.

PQL – practical quantitation limit – The lowest level that can be achieved reliably within specified limits of precision and accuracy during routine laboratory conditions. It is matrix dependent and is simply calculated as a multiple of the MDL. Each compound or element is assigned a multiplier that is contingent upon the behavior of the compound or element during analysis (generally 5 or 10).

RDL – reliable detection limit – A measurement required for risk assessment. It is similar to a PQL but is derived from the MDL by multiplying the MDL by 2.623. “Human Health Risk Assessment Protocol” U.S. EPA, Office of Solid Waste, July 1998.

EDL – estimated detection limit – This detection limit is used for isotope dilution methods only and is the detection limit that is reported for a target analyte that is not detected, or presents an analyte response that is less than 2.5 times the background level. EDLs are different for every sample.

5.3.2 Process Sample Properties

To demonstrate the reproducibility of the analyses of physical/chemical properties (e.g., heating value, ash content, elemental analysis, and total chlorine) in waste feed samples, analyses will include a prescribed number of duplicate analyses. Laboratory standards will be used to demonstrate the accuracy of the analytical methods applied to the project samples.

5.3.3 Process Sample Metals

A system of post-digestion spikes and matrix spikes are performed to provide appropriate and defensible reporting limits for metals in waste feed samples. Spiked samples will be spiked with metal analytes at 3 to 5 times the MDL used for the inductively coupled argon plasma spectroscopy (ICP) for non-mercury (non-Hg) metals and cold vapor atomic adsorption spectroscopy (CVAA) for mercury (Hg) to demonstrate the recovery and reproducibility of the methods.

5.3.4 Process Sample Organics

Matrix spikes are performed to provide appropriate and defensible reporting limits for organics in waste feed, caustic, makeup water, and blowdown samples. Spiked samples will be spiked with POHCs at 3 to 5 times the MDL used for the GCMS to demonstrate the recovery and reproducibility of the methods

5.3.5 Stack Gas VOST

Non-detect results for the volatile POHC are not expected for VOST samples due to the relative rates that the volatile POHC will be present in or will be spiked into the waste feeds. Therefore, performance is not based on the POHC detection limits. Should it be necessary to demonstrate that non-detect values are appropriate, blank spike samples will be performed by the laboratory to demonstrate the recovery and reproducibility of spikes of the POHC applied to the Tenax material of each tube of a VOST tube pair.

Volatile PICs will also be assessed using the VOST. A target list of volatile organic compounds is included in this test program. Compounds on the target compound list will be reported per the standard SW-846 method using laboratory-determined MDLs. In addition to the target list of compounds, all non-target compound list peaks exhibiting a relative response greater than 10% of the nearest internal standard will be tentatively identified using a library search for all SW-846 Method 8260 analytes. TICs will be quantified based on the nearest internal standard and reported.

5.3.6 Stack Gas Dioxin/Furans

Each dioxin and furan 2,3,7,8-congener and tetra- through octa- congener group have sample-specific detection limits (EDLs). When Method 0023A stack gas samples for dioxins and furans are non-detect for a target dioxin or furan analyte, a sample-specific detection limit is calculated for that analyte. This is done by determining the HRGC/HRMS peak height of the noise or interferent in the expected region of the analyte signal. This value is typically multiplied by a factor of 2.5. The resulting EDL signal response value is then used in the sample calculation as if it were a detected value. The result is the estimated sample detection limit.

5.3.7 Stack Gas SVOCs, PAHs, OCP, and PCBs

Semivolatile organic compound (SVOC) PICs, including polynuclear aromatic hydrocarbons (PAHs), organochlorine pesticides (OCP), and polychlorinated biphenyls (PCBs), will be assessed using the Method 0010 sampling train. The sampling train XAD-2 resin traps will be pre-spiked with isotopically labeled surrogate compounds of SVOCs, PAHs, and PCBs. The prepared extracts from the sampling train will be split four ways for the separate analyses and an archive. The relative accuracy of the recovery of the surrogate spikes will demonstrate the required accuracy performance.

A target list of semivolatile organic compounds (SVOCs) is included in this test program. Compounds on the target compound list will be reported per the standard SW-846 method using laboratory-determined MDLs. In addition to the target list of compounds, all non-target compound list peaks exhibiting a relative response greater than 10% of the nearest internal standard will be tentatively identified using a library search for all SW-846 Method 8270 analytes. TICs will be quantified based on nearest internal standard and reported.

The target list of the PAH compounds includes the 20 PAH compounds included in California Air Resources Board (CARB) Method 429. The target list of the OCP compounds includes 26 of the 28 compounds listed in SW846 Method 8081. The two compounds not included for analysis as pesticides are included in the SVOC list. The target list of the PCB compounds includes the 13 dioxin-like (coplanar) PCB compounds and the ten homologue groups (mono- though deca-CB). PAHs and PCBs will be analyzed via high resolution GC/MS (HRGC/HRMS). Like dioxins and furans, when stack gas samples are non-detect for a target PAH analyte, a sample-specific detection limit is calculated for that analyte. This is done by determining the HRGC/HRMS peak height of the noise or interferant in the expected region of the analyte signal. This value is typically multiplied by a factor of 2.5. The resulting EDL signal response value is then used in the sample calculation as if it were a detected value. The result is the estimated sample detection limit.

5.3.8 Stack Gas Metals Samples

Non-detect results may be reported for Method 29 stack gas samples for some metals. The test program includes collection of two blank Method 29 trains, one for a matrix spike sample and one for a matrix spike duplicate sample. The analysis program also includes post-digestion spikes of actual Method 29 samples. The recovery and reproducibility of these spikes and analyses will serve to prove that any non-detect values for Method 29 stack gas samples are valid. The spiked samples will be spiked with metal analytes at 3 to 5 times the MDL used for the ICP and CVAA to demonstrate the recovery and reproducibility of the preparation and analysis methods.

5.3.9 Stack Gas Hexavalent Chromium

Non-detect results may be reported for Method 0061 stack gas samples for hexavalent chromium. The test program includes the analysis of a matrix spike analyzed in duplicate. Additionally all samples are analyzed in duplicate. The recovery and reproducibility of these spikes and sample analyses will serve to prove that any non-detect values for Method 0061 stack gas samples are valid. The spiked samples will be spiked with metal analytes at 1 to 2 times the apparent concentration of the unspiked sample or at 10 times the MDL.

5.3.10 Stack Gas Chloride and Particulate

Non-detect results are expected for Method 26A stack gas chloride. The matrix spike samples of the Method 26A impinger solutions will show that any non-detect values are appropriate. Matrix spikes will be performed at levels 3 to 5 times the MDL used for the ion chromatography method to demonstrate the recovery and reproducibility of the method. Each field sample, calibration standard and QC sample will be analyzed in duplicate. For particulate analyses, duplicate measurements will entail replicate weight determinations to demonstrate reproducibility and consistency, and balance calibration standards will be used to assess analytical accuracy.

5.3.11 Stack Gas Method 0040 VOC Samples

SW-846 Method 0040 includes collection and analysis of Tedlar bag samples of the stack gas for C_1 through C_7 hydrocarbons via gas chromatograph/flame ionization detector (GC/FID) analysis. The GC/FID instrument will be setup and calibrated in the field. Bag samples will be analyzed within seventy-two hours of collection for C_1 through C_7 hydrocarbons. The GC/FID will be calibrated in accordance with Method 0040 procedures before and after analysis of the bag samples. Method blank analysis includes nitrogen Tedlar bag samples. The calibration and blank analysis results will demonstrate the required accuracy performance. Condensate samples recovered from the Method 0040 sampling will be collected in 40 ml VOA vials, topped off with deionized water (for zero head space) and submitted to the laboratory for analysis with 14 days of collection.

5.3.12 Stack Gas Method 0010 TCO/Grav Samples

A variation of the SW-846 Method 0010 sampling train [Modified Method 5 (MM5)] sampling train will be used to sample the stack gas for total semivolatile organic compounds (Boiling Points from 100°C to 300°C) and non-volatile organic compounds (Boiling Points greater than 300°C). The XAD-2 resin traps used in this sampling train do not have isotopically labeled surrogates added before their use in the field.

The sample extraction process also excludes the use of isotopically labeled surrogates. The extracts of the pooled components of the sampling train are used to determine the Total Chromatographable Organics (TCO) via GC/FID analysis in the laboratory. The marker compounds are n-heptane and n-heptadecane because their boiling points are 98°C and 302°C, respectively. The non-volatile organics are determined by a gravimetric procedure known as Grav from the same pooled extracts of the Method 0010 train components as the semivolatile organic components. Because there are no isotopically labeled surrogates used within the sampling and analysis regime of this method, the only accuracy measurement for this sampling method is the blank train samples prepared and analyzed in the same manner as the actual field samples. The TCO/Grav blank train results will be used to blank correct the test TCO/Grav results.

5.3.13 Stack Gas Method 5 PSD

A method 5 train with polycarbonate or acetate filter is used to collect samples for particle size distribution via scanning electron microscope (SEM) analysis. The size distribution of the particulate captured on the filter is determined by an actual count of particles of different sizes found on the filter.

5.4 COMPLETENESS

Data completeness represents the percentage of valid data collected from the total number of valid tests conducted. Completeness is usually expressed as a percentage and calculated based on the number of samples reaching the laboratory for analysis. The completeness objective for the test will be met (100% completeness) if three valid test runs are obtained for each test condition. Samples resulting from test runs that are judged invalid based on field performance indicators or aborted runs will not be submitted to the laboratory for analysis. Because the possibility exists that a sample may be lost or broken, the data from each individual analytical parameter may not be 100 percent complete for all test runs. The impact of any occurrence of sample loss will be assessed with regard to the objective of obtaining valid runs and will be discussed in the test report. The completeness objective of this test program is to generate sufficient data for the regulatory agencies to judge the performance of the system. An overall completeness objective is to meet at least 90% of the total data quality objectives established in the QAPP. Contingency samples have been incorporated into the sampling design in an effort to gather complete data. Archive samples of most feeds and residues are collected as a contingency for breakage or analytical difficulties.

5.5 REPRESENTATIVENESS, SENSITIVITY AND COMPARABILITY

The sampling procedures chosen for the test are, wherever possible, approved EPA or American Society for Testing Materials (ASTM) sampling methods that are typically employed on incinerator tests. The use of standard sampling methods affirms sample representativeness.

Sensitivity for this test is a function of the sample matrix, the sample size, and the analytical detection limit. The sample sizes chosen for each sample matrix are such that the collected sample is greater than the sample volume/mass required for each analytical method to obtain an acceptable quantitation limit for the project. Calculations have been provided as part of the PDT plan to indicate that the selected sample sizes and analytical methods are appropriate for critical test determinations.

Use of standard, approved sampling and analysis methods, standardized data reduction procedures, and QC samples will provide data that is technically defensible and is comparable from test run to test run, test condition to test condition.

6.0 SAMPLING AND MONITORING PROCEDURES

6.1 GENERAL

The objectives of this test program are the collection of representative feed, process, and stack gas samples that will demonstrate compliance of the RF system with the applicable performance and emissions standards, and provide emissions data for the post-test risk assessment conducted under RCRA Omnibus authority [40 CFR 270.32(b)(2)]. To meet these objectives requires minimizing the potential sources sample contamination or bias imparted to the samples by the sampling equipment, ambient conditions, handling, and preservation. The test program samples will be collected using the methods summarized in Table 6-1. The total numbers of field samples expected to be generated during the WCAI system tests are summarized in Tables 6-2. Sampling procedures are included in Attachment A to the Performance Demonstration Test Plan and are incorporated here by reference.

Guidelines followed to determine sampling equipment to be used, sampling points, and the frequency at which samples are to be taken are presented in Section 5.0 of the PDT plan, and are incorporated here by reference. The reference sources for the standard sampling method references include: Appendix A to 40 CFR 60, *Test Methods and Procedures, New Source Performance Standards*, 40 CFR 60 (EPA); *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition, 1986 and updates (SW-846); and the *American Society for Testing and Materials* (ASTM) Annual Book of ASTM Standards. Regulatory observer approval will be requested if significant deviations from planned procedures are encountered during the testing.

All stack sampling equipment and glassware will be prepared prior to the test according to the method specifications. Following each run, the samples will be recovered from the trains. The sample recovery procedures include prescribed rinses of the trains, which serve a dual purpose of sample recovery and decontamination of the train in preparation for the next run. Rinses that are not included in the sample recovery will be placed into a waste solvent container and disposed of by WCAI.

Process samples will be collected using dedicated sampling equipment (scoops, jars, etc.) at each sampling location, thus eliminating the potential for cross contamination from one sample matrix to another. New sampling containers are used for each test run. If the same equipment will be used for more than one run, the equipment will be decontaminated by thorough washing with detergent, water and, any additional rinses required by the specified sampling and analytical protocol for which the equipment will be used. Any decontamination solution generated, will be collected by the facility operators for proper disposal.

During the test program, the RF system will be operated and tested at the conditions specified in the respective PDT plan. The following samples will be collected during the test:

- Feed Samples:
  - Spent activated carbon
- Process Samples:
  - Makeup water
  - Caustic
  - Scrubber blowdown
  - POTW discharge
- Stack Gas Samples
  - SW-846 Method 0030 VOST for Volatile POHCs and PICs
  - SW-846 Method 0023A for PCDD/PCDFs
  - EPA Method 29 for Metals
  - EPA Method 0061 for Hexavalent Chromium
  - EPA Method 26A for Particulate, HCl and Cl_2
  - SW-846 Method 0010 for SVOC and PICs and OCP
  - SW-846 Method 0010 for PAH and PCB
  - SW-846 Method 0010 for TCO/Grav Organics
  - SW-846 Method 0040 for Total Volatiles
  - EPA Method 5for Particle Size Distribution (PSD)
  - Temporary CEMS for THC (EPA Method 25A)
  - Installed CEMS for Carbon Monoxide and Oxygen

Sample tracking is documented using unique sample numbering applied to every sample (refer to Figure 7-5), completed sample collection forms, completed chain of custody (COC) forms, completed request for analysis (RFAs) forms, and sample collection checklists (Figures 7-1, 7-2, and 7-3).

6.2 FIELD QUALITY CONTROL SAMPLES

Field QC samples will be collected during the test to provide an indication of quality assurance for the test samples. The field QC samples include: spiked resin blanks for VOST, Method 0023A and Method 0010 for SVOC and PICs, PAH, and PCB; field and trip blanks for VOST; reagent blanks for all sampling trains; and blank trains for Method 29, Method 0023A, Method 0010 for SVOC and PICs, PAH, OCP, and PCB, and Method 0010 TCO/Grav. Table 6-2 includes the field QC samples that will be collected.

6.2.1 Spiked Resin Blanks

During the preparation of the Tenax and Tenax/Charcoal tubes sets for the test program, two VOST Tenax and Tenax/charcoal resin tube pairs will be spiked with standard EPA contract laboratory program (CLP) surrogate spike compounds. These samples will be analyzed to determine by the VOST tube preparation laboratory to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

Two XAD-2 resin traps prepared for the Method 0010 SVOC/OCP and PAH/PCB sampling trains will be spiked with sampling surrogates and internal standards. These samples will be extracted and analyzed for SVOCs, PAHs, OCP, and PCBs by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

Two XAD-2 resin traps prepared for the Method 0023A sampling train will be spiked with sampling surrogates and internal standards. These samples will be extracted and analyzed for dioxins and furans by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

Two unspiked XAD-2 resin traps prepared for the Method 0010 TCO/Grav train will be extracted and analyzed by the XAD-2 trap preparation laboratory to demonstrate the resin is free of background contamination.

6.2.2 Feed and Process Samples

Standard methods, procedures, and dedicated sampling equipment will be used for the collection of process samples associated with this test program. The Process Sampling Coordinator monitors the process sampling during the testing to assure that proper documentation is completed and that adherence to prescribed sampling procedures is observed. Sample tracking is documented using the sample numbering system (Figure 7-5), completed RFAs and COCs, the field sampling record, and sample collection checklist (Figures 7-1, 7-2, and 7-3).

6.2.3 Stack Gas Samples

During the test program, the Stack Sampling Coordinator and the Sample Custodian are responsible for monitoring the sampling team's adherence to the standard stack sampling procedures, especially sampling train preparation; leak checks and recoveries (including blank trains); and reagent, field, and trip blanks. The Stack Sampling Coordinator is responsible for operation and recovery of the stack sampling equipment and stack gas samples. The Sample Custodian is responsible for preparing the stack gas samples for shipment to the laboratory. Sampling train calibration procedures are discussed in Section 8.0.

EPA Methods 1 and 2 will be used to determine the number and location of sampling traverse of isokinetic sampling locations. Documentation of the Methods 1 and 2 will be included in the stack sampling report.

During each test run, Tedlar bag samples of stack gas will be collected for determining the stack gas carbon dioxide and oxygen concentrations using EPA Method 3 (Orsat Analysis). The stack gas carbon dioxide and oxygen concentrations are used to determine the stack gas molecular weight. The Tedlar bag samples may be collected from the exhaust of one of the isokinetic sampling trains or using a separate impinger and vacuum pump setup. Carbon dioxide and oxygen calibration gases will be used as reference standards for the Orsat apparatus. As an alternate to Orsat analysis, calibrated CEMS may be used for oxygen and carbon dioxide determination.

Stack gas moisture content will be determined for each isokinetic sampling train via EPA Method 4 (sampling train moisture gain). Isokinetic sampling trains silica gel impingers will be filled with fresh, dry indicating silica gel at the beginning of the test program. During the sampling train recovery process, and subsequent test runs, each indicating silica gel impinger will be inspected prior to reuse to verify that sufficient capacity remains for moisture absorption during the next test run. Silica gel more than 50% utilized will be discarded and the impinger recharged with fresh dry indicating silica gel.

6.2.3.1 VOST

The VOST will be used to sample stack gas for the selected volatile POHC and volatile PICs. The VOST data will be used to assess POHC DRE and volatile PIC emissions. The VOST sampling apparatus will be inspected and leak checked prior to each test run. Four pairs of VOST tubes will be used during each sampling run to sample a nominal 20 liters of stack gas per tube set. Three of the four pairs will be analyzed by Method 5041A/8260B. The fourth set pair will serve as an archive set in the event of sample breakage during shipping and handling. During the analysis, the stack gas and VOST audit Tenax and Tenax/charcoal tube sets will be analyzed separately to assess breakthrough. Field and trip blank Tenax and Tenax/charcoal tube sets are also analyzed separately.

6.2.3.2 Method 0010 for SVOC and PICs, PAHs, OCPs, and PCBs

Two Method 0010 sampling trains will be used to sample stack gas semivolatile organic compound (SVOC) PICs including polynuclear aromatic hydrocarbons (PAHs), organochlorine pesticides (OCP), and polychlorinated biphenyls (PCBs). During each test run, the Method 0010 sampling trains will be assembled and leak checked. The sampling train will be operated to sample a minimum of three dry standard cubic meters of stack gas during each sampling run. The sampling time to achieve the target sample volume will depend upon the probe nozzle size and sampling rate. However, sampling rate will not exceed one dry standard cubic foot per minute. At the end of each run, the sampling train will be disassembled and all train samples collected.

The Method 0010 sampling train components will be prepared for analysis following SW-846 Method 3542. Method 3542 results in three separate fractions for analysis of the sampling train. Surrogates compounds discussed below are applied to each fraction.

The first fraction, the front-half fraction, consists of the solvent probe rinses and the particulate filter. The front half samples will be combined. Surrogate compounds for the SVOCs, PAHs, OCPs, and PCBs are spiked directly onto the filter before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride.

The second fraction consists of the XAD-2 resin and condenser rinse. The XAD-2 resin and condenser rinse samples will be combined. Surrogate compounds for the SVOCs PAHs, OCPs, and PCBs are spiked directly onto the XAD-2 resin before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride.

The third fraction consists of the condensate impinger catch and rinses. The condensate impinger catch is volumetrically or gravimetrically measured in the field, and this information is added to the stack sampling data sheet to calculate the moisture content of the stack gas. In the laboratory, the condensate impinger sample is placed in a separatory funnel and surrogate compounds for the SVOCs, PAHs, OCPs, and PCBs are added. The sample is then pH adjusted to acid (pH<2) or basic (pH>11), and is then separatory funnel extracted using methylene chloride and deionized water. The methylene chloride extract layer is then removed and fresh methylene chloride added to the separatory funnel. The methylene chloride/water separatory extraction is then repeated with the pH adjusted to either acid or basic, depending on the original adjustment. The second methylene chloride extract layer is then removed and combined with the earlier acid or basic methylene chloride extract.

For SVOCs, four types of spiking materials will be applied to the Method 0010 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the SVOCs found using the Method 0010 sampling method.
- Surrogate Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.

- Semivolatile Internal Standard Compounds - These compounds are applied to the sample extract just prior to GC/MS analysis. These compounds applied to the samples are used to determine if the continuing calibration internal standards are still appropriate to use to calculate the associated compound concentrations.
- Matrix Spike Compounds - These compounds are spiked onto the condensate portion of the Method 0010 samples and onto a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and blank resin sample before GC/MS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

For OCPs, three types of spiking materials will be applied to the Method 0010 sampling train samples:

- Surrogate Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.
- Organochlorine Pesticides Internal Standard Compounds - These compounds are optional and may be applied to the sample extract just prior to GC/MS analysis. These compounds applied to the samples are used to determine if the continuing calibration internal standards are still appropriate to use to calculate the associated compound concentrations.
- Matrix Spike Compounds - These compounds are spiked onto the condensate portion of the Method 0010 samples and onto a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and blank resin sample before GC/MS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.

For PAHs five types of spiking materials will be applied to the Method 0010 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the PAHs found using the Method 0010 sampling method.
- Internal Standard Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. These standards are used to measure the concentration of the analytes and surrogates.
- PAH Recovery Standards- Isotopically labeled compounds applied to the Soxhlet extracts just before HRGC/HRMS analysis. Recovery standards are added to the sample are used to estimate the recovery of the internal standard. Recovery of the internal standard is an indicator of the overall performance.
- Matrix Spike Compounds - These compounds are spiked only to the condensate portion of the Method 0010 samples and a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and a blank XAD resin sample before HRGC/HRMS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.
- Alternate Standard – This compound is added to the impinger prior to extraction. The alternate standard is used to estimate extraction efficiency for the PAH impingers.

For PCBs five types of spiking materials will be applied to the Method 0010 sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD-2 resin tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the PCBs found using the Method 0010 sampling method.
- Isotopically Labeled Analogs - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. These standards are used to measure the concentration of the analytes and surrogates. Concentrations for compounds isotopically labeled analogs added to the sample are determined using the isotope dilution technique.
- PCB Cleanup Standards- Cleanup standards are added after extraction and just prior to clean-up to assess the clean-up of the sample.
- Matrix Spike Compounds - These compounds are spiked only to the condensate portion of the Method 0010 samples and a blank XAD resin. The matrix spike compounds are spiked onto an aliquot of the Method 0010 condensate and a blank XAD resin sample before HRGC/HRMS analysis. Recovery of the spikes provides an indicator of method accuracy for the sample matrix.
- PCB Internal standards – These compounds are applied to the sample extract just prior to GC/MS analysis. These compounds applied to the samples are used to determine if the continuing calibration internal standards are still appropriate to use to calculate the associated compound concentrations. Concentrations for compounds without isotopically labeled analogs are determined using the internal standard method.

This test program includes target lists of SVOCs PAHs, OCPs, and PCBs for the Method 0010 SVOC PIC sampling. Commensurate with the procedures allowed under at 40 CFR 63.1208(b)(1)(iii) for the Method 0023A analysis for dioxin/furan congeners, any target SVOC, OCP, or PAH compound that is non-detect in all three analysis fractions of the Method 0010 sampling train will be counted as zero in determining the emissions data for use in the post-test risk assessment conducted under RCRA Omnibus authority [40 CFR 270.32(b)(2)]. For any of the non-target SVOC compounds tentatively identified using a library search for all SW-846 Method 8270 analytes not found during all three runs of the test, the tentatively identified compounds (TICs) will be averaged with zeros for the runs where they are not identified.

6.2.3.3 Method 0023A

A Method 0023A sampling train will be used to sample stack gas dioxin and furans. During each test run, the Method 0023A sampling train will be assembled and leak checked. As required by 40 CFR 63.1208(b)(1)(ii), the Method 0023A sampling train will be operated a minimum of 180 minute (3 hours) to sample a minimum of 2.5 dry standard cubic meters of stack gas during each sampling run. At the end of each run, the sampling train will be disassembled and all train samples collected. In accordance with 40 CFR 63.1208(b)(1)(iii), any dioxin/furan congener that is non-detect will be counted as zero in determining compliance.

The Method 0023A sampling train front half and back half components are prepared and analyzed as two separate fractions. Surrogates compounds discussed below are applied to each fraction.

The first fraction, the front-half fraction, consists of the solvent probe rinses and the particulate filter. The front half samples will be combined. Internal standard compounds for the PCDDD/PCDFs are spiked directly onto the filter before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride followed by 18 hours of extraction using toluene. The methylene chloride and toluene front-half extracts are combined and then blown down. Recovery standards are then added prior to the final concentration for HRGC/HRMS analysis via SW-846 Method 8290.

The second fraction consists of the XAD-2 resin and condenser rinse. The XAD-2 resin and condenser rinse samples will be combined. Internal standard compounds for the PCDD/PCDFs are spiked directly onto the XAD-2 resin before the Soxhlet extraction. The sample is then Soxhlet extracted for 18 hours using methylene chloride followed by 18 hours of extraction using toluene. The methylene chloride and toluene back-half extracts are combined and then blown down. Recovery standards are then added prior to the final concentration for HRGC/HRMS analysis via SW-846 Method 8290.

Three types of spiking materials will be applied to the Method 0023A sampling train samples:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD tube preparation and prior to stack sampling. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the dioxin and furans found using the Method 0023A sampling method.
- Isotope Dilution Internal Standard Spikes - Isotopically labeled compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling and analysis by the laboratory.
- Dioxin and Furan Recovery Standards- Isotopically labeled compounds applied to the Soxhlet extracts just before GC/MS analysis. These compounds provide the relative response factors which are used to calculate analyte concentrations.

6.2.3.4 Method 29

An EPA Method 29 sampling train will be used to sample stack gas for the project target metals. Samples are subjected to acid digestion using nitric and hydrofluoric acid in either a parr bomb or microwave pressure relief vessel. Non-mercury metals will be analyzed by SW-846 Method 6010B [inductively coupled argon plasma spectroscopy (ICP or ICAP)] or 6020 [inductively coupled argon plasma spectroscopy/mass spectroscopy (ICP/MS or ICAPMS)]. Mercury will be analyzed for by SW-846 Method 7470 [cold vapor atomic absorption spectroscopy (CVAA or CVAAS)]. Accuracy and precision are measured though the use of a matrix spike and matrix spike duplicate. Two blank trains are collected and spiked to provide the MS/MSD analysis. The five Method 29 sampling train fractions will undergo seven separate analyses as follows:

- The nitric acid probe rinse and the particulate filter will be combined and digested in the laboratory as the front half composite sample and analyzed for Hg and the non-Hg target metals.
- The condensate knockout impinger (impinger 1) and the HNO_3/H_2O_2 impingers (impingers 2-3) catches will be digested in the laboratory and analyzed for Hg and the non-Hg target metals.
- The empty impinger (impinger 4) catch will be digested in the laboratory and analyzed for Hg.
- The $KMnO_4/H_2SO_4$ impinger (impingers 5-6) catches will be digested in the laboratory and analyzed for Hg.
- The 8N HCl rinse of impingers 5-6 will be digested in the laboratory and analyzed for Hg.

6.2.3.5 Method 0061

A Method 0061 sampling train will be used to sample for hexavalent chromium during Test 2. Stack samples are filtered, preconcentrated and analyzed directly using ion chromatography following SW846 Method 7199. Accuracy and precision are determined through the use of duplicate analysis, laboratory control samples, and matrix spikes.

6.2.3.6 EPA Method 26A

An EPA Method 26A sampling train will be used to sample for particulate, HCl and Cl_2, during each test run. The stack gas is sampled by bubbling the gas through impingers containing 0.1N H_2SO_4 (acid) and 0.1N NaOH (basic) solutions in series. In the acid impinger solution, HCl gas is captured. Any Cl_2 passes through to the acid impinger and is captured in the basic impinger solution. The chloride concentrations of the acidic and basic impinger samples are analyzed separately for chloride ion, and are reported as HCl and Cl_2 catches respectively. During the sampling train recovery, the basic Cl_2 catch is preserved as by addition of 0.008% sodium thiosulfate ($Na_2S_2O_3$). The sodium thiosulfate preserves the Cl_2 sample by converting any Cl_2 captured as hypochlorite ion (OCl^-) to the more stable chloride ion (Cl^-). Precision for these samples is determined through the use of duplicate analyses of all calibration standards, all QC samples and all field samples. Accuracy is determined by matrix spike/ matrix spike duplicate analyses. Additional matrix specific quality control is provided by separate matrix specific calibrations being analyzed prior to sample analysis.

The stack gas particulate emissions are determined by weighing the tare weighted particulate filter to determine the differential weight of the particulate collected by the Method 26A sampling train. Samples are dried to a constant weight to the nearest 0.1 mg. Constant weight shall mean a difference between two consecutive weighings of no more than 0.5 mg difference or more than 1 percent of the total weight less the tare weight. This differential weight is added to the weight of the residue remaining after evaporation of the acetone probe and filter holder rinses. The reported particulate catch is the sum of the particulate filter differential weight and the probe rinse residue weight.

6.2.3.7 Method 0010 for TCO/Grav

A Method 0010 sampling train will be used to sample stack gas total chromatographable organics (TCO and gravimetric organics (Grav). During each test run, the Method 0010 sampling train will be assembled and leak checked. The sampling train will be operated to sample a minimum of three dry standard cubic meters of stack gas during each sampling run. The sampling time to achieve the target sample volume will depend upon the probe nozzle size and sampling rate. However, sampling rate will not exceed one dry standard cubic foot per minute. At the end of each run, the sampling train will be disassembled and all train samples collected.

In the laboratory, the TOC/Grav front and back half rinses, particulate filter and XAD-2 resin are combined and Soxhlet extracted with methylene chloride. The final pooled extract volume must be no less that 5 mL. Since the extracts for total organics determinations are analyzed by GC/FID and gravimetric techniques, no sampling surrogate, isotopically-labeled standards, or internal standards associated with GC/MS analysis (Method 8270) are added to the extractors or sample extracts. The combined methylene chloride extracts are split into four portions and used as follows:

- One portion of the extract is used for semivolatile organic analysis using TCO GC/FID analysis according to EPA Guidance for Total Organics. Details of the method are described in Appendix C of the Guidance.
- Two portions of the methylene chloride extract are used for nonvolatile organic analysis using gravimetric mass (Grav) method according to EPA Guidance for Total Organics. Details of the method are described in Appendix D of the Guidance.
- The final portion is used as an archive sample.

6.2.3.8 Method 5 for PSD

A method 5 train will be used to sample for particle size distribution (PSD). . Stack gas is sampled isokinetically to collect particulate matter on a polycarbonate or acetate filter. The stack sampling train is operated for a time selected by the sampling team to obtain enough particle mass (without overloading the filter) for particle size determination. The filter is carefully recovered from the sampling train, placed into a petri dish, and sealed. The is then analyzed by a SEM to determine the particle size distribution.

6.2.4 Blank Trains and Reagent Blanks

Blank train samples are the samples recovered from sampling trains that have been assembled and charged with all the required chemical reagents and collection media in the same manner as the sampling trains used to sample the stack gases. The sampling trains are leak checked and heated to temperature in a location near the stack. The sampling train remains sealed at the stack location for a period equivalent to the length of time the corresponding sampling train is operated during the test run. The blank train is then recovered in the same way that actual stack gas sampling trains are recovered. The recovered blank train components are labeled as blank train samples and submitted for analysis with the actual stack gas train samples. The results of the blank train samples provide an indication of possible contamination introduced to the samples by reagents, glassware, sampling environment, and sampling recovery. The blank train samples for the stack sampling trains used during this test program will be collected as summarized in Tables 6-2. For this test program one baseline test, and up to two optional, additional tests, for a maximum of nine test runs total, two Method 29 blank trains, one for a matrix spike sample and one for a matrix spike duplicate sample, will be collected. One each blank train of the each the Method 0010 SVOC/OCP, Method 0010 PAH/PCB, Method 0023A, and Method 0010 TCO/Grav trains will be collected during the testing.

Reagent blanks are samples of the reagent source solvents, solutions, and other media used in stack sampling. Reagent blank samples for the Method 26A, Method 29, Method 0023A, Method 0010 SVOC/OCP, Method 0010 PAH/PCB, and Method 0010 TCO/Grav sampling trains used during this test program will be collected as summarized in Table 6-2.

6.2.5 Field Blanks

Field blanks are sampling media that are handled at the sampling location in the same manner as the actual test samples. However, these media are not used to collect stack gas samples. The field blank samples will be collected and analyzed to demonstrate that the sample handling procedures do not expose the samples to contaminants. This test program includes collecting one pair of VOST tubes as field blanks once during each test run (three samples total). Each field blank VOST tube pair is opened in the field by the VOST operator during the sampling run and are allowed to remain open for a period equivalent to the time required for a tube pair change-out during testing. The field blank pair is then sealed up, labeled and handled in the same manner as other VOST samples. The compounds found in field blanks reflect exposure to field fugitives, laboratory contaminants and resin degradation products, and are used to assess any contamination that can impact test results.

6.2.6 Trip Blanks

Trip blanks are similar to field blanks in that they are used to assess contamination resulting from sample shipment. With each shipment of VOST samples from the test site to the laboratory, a pair of VOST tubes that have remained sealed as shipped from the laboratory to the field to be used as trip blanks. Additionally, pair of volatile organic analysis (VOA) vials filled with deionized (DI) water is included with the VOST samples shipped from the test site back to the laboratory. The trip blank and DI water analyses demonstrate that the samples are not exposed to contamination during transport from the field to the laboratory.

7.0 SAMPLE HANDLING, TRACEABILITY, AND HOLDING TIMES

7.1 SAMPLE CUSTODY

A sample will be considered to be in the custody of a person if it is in his or her possession, in his or her sight, or secured by that person in an approved location accessible only to authorized personnel.

The analytical laboratory will prepare the sampling media and sample reagents according to the specifications of the methods as described in the PDT plan and will ship them to the site in sealed containers.

During the test, once the samples are transferred from the sampling technician to the Sample Custodian, sample custody becomes the responsibility of the Sample Custodian until the samples arrive at the analytical laboratory. When overnight couriers are utilized, the air bill will serve to document the transfer of custody from the Sample Custodian to the courier. The courier's air bill becomes part of the chain of custody (COC) record. Upon transfer of the samples from the courier to the analytical laboratory, sample custody will be maintained by the analytical laboratory performing the analyses. Samples for organic analysis (VOST, Method 0023A, POHC, etc.) will be kept on ice ($4 \pm 2^{o}C$) and shipped to the analytical laboratory in sealed, insulated shipping containers (ice chests). All ice on shipped samples shall be triple bagged in ziplock bags to prevent leakage of water during shipping. Samples not requiring chilling particulate, chloride, metals, properties, etc.) may be shipped in sealed, insulated shipping containers (ice chests) without ice. **If dry ice is used to preserve organic samples during shipping, the shipment packaging and placarding shall be conducted in strict accordance with the International Air Transport Association (IATA) regulations.**

Collected samples will be shipped from the site to the laboratory in sealed containers with chain of custody (COC) and request for analysis (RFA) forms. Example COC and RFA forms are presented as Figures 7-1 and 7-2 respectively. Prior to shipping any samples, the condition of these samples will be documented on the chain of custody by the Sample Custodian. Conditions to be verified and documented on the COC include but are not limited to the following: sample temperatures for organic analyses samples (measurement will be taken using the temperature blank), condition of all containers, level of sample within all containers (to be marked on the outside of the container), and type of packing material used. RFAs will be checked against the COCs to verify there is a RFA for every sample being shipped. If samples are shipped by overnight courier , the Sample Custodian shall make three photocopy sets of all COCs and RFAs before shipping the samples. One copy will be retained by the Sample Custodian, and one copy each will be provided to the Test Coordinator and the QAO.

Upon receipt, the shipping containers will be opened by the Laboratory Analysis Coordinator or his designee and inspected. The receiver will verify that the container contents correspond with the COC. Any damage to the contents of the shipping container or deviations from the original shipment documents will be noted on the COC and the receiver will accept custody for the shipment by an exchange of signatures with the delivering agent.

Upon receipt of samples at the laboratory, the sample shipping containers will be opened and all sample containers inspected. A labeled temperature blank (labeled VOA vial with water) will be shipped in every container with samples for organic analysis expressly for the purpose of determining sample temperatures. **The Laboratory Analysis Coordinator or designee will, immediately upon opening the sample packaging, open the temperature blank and measure the temperature of the water inside the temperature blank using a thermometer. This temperature will be recorded on the COCs and any applicable laboratory documentation (sample receipt log).** Containers will then be secured in a location accessible only to authorized personnel. Samples for organic analysis shall be secured in refrigerated sample storage. The COC forms are used specifically to track the samples. To provide specific instructions to the analysts, the RFAs will accompany the respective COCs.

Transfer of custody to and within the analytical laboratory is addressed in the Laboratory's QA Manual. Upon completion of analysis, samples will be maintained at the laboratory under chain of custody until they are released for proper disposal.

7.2 SAMPLE LABELING

An example sample label format is presented in Figure 7-4. Each sample container will be labeled to show the source of the sample as WCAI; the project identification; sampler's initials; laboratory to which the sample will be shipped; an unique alphanumeric sample number; date and time; sample description; test number; and run number. If a single sample requires multiple containers, the number of the container and the total number of containers will be noted on the label.

Project samples will be tracked via the assigned unique alphanumeric sample numbers. The sample number will appear on the sample label, the RFA and the COC. The alphanumeric system of sample identification for this project is presented in Figure 7-5. The numbering system presented will result in unique numbers being assigned to every sample.

7.3 PROCESS SAMPLE COLLECTION FORMS

While a process sample is being taken in the field, the sampling technician will complete a field sampling record. An example field sampling record is presented as Figure 7-3. The field sampling record will be filled out in its entirety for every sample. This will provide information to be used in the final report. The sampling technician shall provide the completed field sampling record and COC form to the Sample Custodian.

7.4 SAMPLE COLLECTION CHECKLIST

Prior to start of testing, a master list of the samples required for the test will be compiled by the Sample Custodian or QAO. This list will identify the samples by their assigned unique alphanumeric sample numbers (refer to Figure 7-5) and the analytical test(s) required. As field samples are acquired and routed through the Sample Custodian, the samples will be checked off against the master list to ensure that all of the appropriate samples have been taken.

7.5 REQUEST FOR ANALYSIS/CHAIN OF CUSTODY

The Sampling Technician and Sample Custodian will complete the COC and RFA forms for every sample. Each sample may consist of several sub-samples. Each individual component of the sample will be listed separately on the COC with its own unique alphanumeric sample identification number. The samples will be preserved as needed and secured in a shipping container by the sampler and must remain in his or her possession until it is presented to the Sample Custodian. The Sample Custodian will secure the samples in a location accessible only to authorized personnel until custody is transferred to a courier for delivery to the laboratory.

Each sample container will be clearly identified using standard container labels. It is imperative that information on the COC form, RFA form and the container label match in every respect. Example COC and RFA forms are presented in Figures 7-1 and 7-2. The label format is shown in Figure 7-4. Planned sample identification codes are shown in Figure 7-5. An individual trained in Federal Department of Transportation (DOT) and International Air Transport Association (IATA) regulations will package the samples for overnight courier shipment to be in compliance with the applicable portions of these regulations.

7.6 SAMPLE PRESERVATION

Table 7-1 shows the appropriate containers, preservation, and holding times for all samples to be collected during the test.

The location of the VOST sample holding area is of special importance. The sample containers for volatiles will be stored in a clean area separate from the sample preparation area. High concentration volatile organic samples (recovered elements of other sampling trains, waste feed samples, etc.) will be segregated to prevent inadvertent contamination of the VOST samples. The VOST tube sample pairs will be preserved before and after sampling by placing them on triple bagged ice in a dedicated sample cooler. To preclude contamination from solvents and process samples, VOST samples will be shipped on triple bagged ice in dedicated shipping containers separate from all other test samples.

XAD-2 traps for the Method 0023A, Method 0010 SVOC/OCP, Method 0010 PAH/PCB, and Method 0010 TCO/Grav trains will be preserved before and after sampling by placing them on triple bagged ice in a dedicated sample cooler. The balance of the Method 0023A, Method 0010 SVOC/OCP, Method 0010 PAH/PCB, and Method 0010 TCO/Grav train sample components will be preserved after sample collection by placing them on triple bagged ice in a dedicated sample cooler. Other organic analysis samples (e.g., process samples for POHC) will be preserved after sample collection by placing them on triple bagged ice in a dedicated sample cooler.

For non-organic analysis samples (particulate, chloride, metals, properties, etc.), sample preservatives (if applicable, refer to Table 7-1) will be used as required by the target analyte. These samples will be stored in dedicated sample coolers. These samples do not require chilling on ice or refrigeration.

8.0 SPECIFIC CALIBRATION PROCEDURES AND FREQUENCY

The objective of this section is to assure that process instrumentation, gas sampling equipment, and analytical instruments are performing properly before conducting the test and analyzing samples. Equipment and instruments used to generate data for determining compliance with performance requirements or to establish quantitative allowable operating limits will be calibrated according to the manufacturer's instructions, prior to and/or during the test as necessary.

The calibration procedures are separated into groups according to the personnel who will perform them. WCAI operations personnel will calibrate the process instruments. Stack sampling equipment will be calibrated by the stack sampling contractor and analytical instruments will be calibrated by the contracted laboratory personnel. The calibration procedures for process instrumentation stack gas sampling, and laboratory analytical instruments are described in the following subsections.

8.1 PROCESS INSTRUMENTATION

Prior to the start of testing, the parameter continuous monitoring system (CMS) (thermocouples, flow meters, pressure transducers, etc.) and the continuous emission monitoring system (CEMS) (installed CO and O_2 monitors) will be calibrated in accordance with the facility standard operating procedures.

During testing, the installed carbon monoxide and oxygen CEMS, the monitors will be calibrated daily. The zero and span checks will be considered a verification of the data quality from these monitors.

CMS and CEMS data will be reported on 1-minute intervals and will be archived in the CMS data acquisition system.

8.2 STACK SAMPLING EQUIPMENT

Sampling equipment is calibrated according to the criteria specified in the reference method being employed. In addition, the guidelines set forth in the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods (EPA-600/4-77-027b) will be followed. Dry gas meters, orifices, nozzles, pitot tubes, etc. are calibrated in accordance with this document. The range of the calibration is specified for all environmental measurements to encompass the range of probable experimental values. This approach ensures that all results are based upon interpolative analyses rather than extrapolative analyses.

Calibrations are designed to include, where practical, at least three measurement points evenly spaced over the range. This practice minimizes the probability that false assumptions of calibration linearity will be made. In addition it is common practice to select, when practical, at least one calibration value approximating the levels anticipated in the actual measurement. Typically, calibration frequency is dictated by the need to demonstrate the stability of the calibration value over the course of measurements. Calibrations are made both pre- and post-test to accomplish the demonstration of stability.

Following the test program, calibrations are checked on all relevant items of sampling equipment to ensure the validity of data collected in the field. New items for which calibration is required are calibrated before initial field use. Equipment whose calibration status may change with use or time is inspected in the field before testing begins and again upon return from each field use. When an item of equipment is found to be out of calibration, it is repaired and recalibrated or retired from service. All equipment is periodically recalibrated in full, regardless of the outcome of these regular inspections.

Data obtained during calibrations are recorded on standardized forms, which are checked for completeness and accuracy by management personnel. Data reduction and subsequent calculations are performed using standard procedures, and are computerized where appropriate. Calculations are checked at least twice for accuracy. Copies of calibration forms are included in the test or project reports.

Emissions sampling equipment requiring calibration include pitot tubes, pressure gauges, thermometers, dry gas meters, and barometers. The following sections elaborate on the calibration procedures for these specific equipment items.

8.2.1 Pitot Tubes

All Type S pitot tubes, whether separate or attached to a sampling probe, are inspected in accordance with the geometry standards contained in EPA Method 2. All Type S pitot tubes are calibrated over an eight-point range with a wind tunnel. A calibration coefficient is calculated for each pitot tube.

Each pitot tube is inspected visually upon return from the field. If a visual inspection indicates damage or raises doubt that the pitot remains in accordance with the EPA geometry standards, the pitot tube is first calibrated, then repaired and recalibrated. The acceptance limits are listed in Table 8-1.

8.2.2 Differential Pressure Gauges

Some meter consoles are equipped with 10-inch water column (W.C.) inclined-vertical manometers. Fluid manometers do not require calibration other than leak-checks. Manometers are leak-checked in the field prior to each test series and again upon return from the field.

8.2.3 Digital Temperature Indicator

One digital temperature indicator is used to determine the flue gas temperature, probe temperature, oven temperature, "train temperature" and dry gas meter temperature. The digital temperature indicator is calibrated over a seven-point range (32°F-450°F) using an ASTM mercury-in-glass thermometer as a reference. The calibration is acceptable if the agreement is within ±2% or 2°F from 50°F-180°F.

8.2.4 Dry Gas Meter and Orifice

A calibrated wet test meter is used to calibrate the dry gas meter and orifice. The full calibration procedure is used to obtain the calibration factor of the dry gas meter. Full calibrations are performed using a calibrated wet test meter as a reference standard.

8.2.4.1 Dry Gas Meter

Each metering system receives a full calibration at the time of purchase and quarterly. Upon request, a post-test calibration can be performed after each field use. If the calibration factor deviates by less than five percent from the initial value, the test data are acceptable. If it deviates by more than 5%, the meter is recalibrated and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

EPA Method 5 requires another full calibration anytime the post-test calibration check indicates that the calibration factor has changed by more than 5%. Standard practice is to recalibrate the dry gas meter quarterly and check the orifice calibration during and after each field use.

8.2.4.2 Orifice

An orifice calibration factor is calculated for each of the eighteen flow settings during a full calibration. The arithmetic average of the values obtained during the calibration is used.

8.2.5 Barometer

Each field barometer is adjusted before each test series to agree within ±0.1 inches of a reference aneroid barometer. The reference barometer is checked against the station pressure value (corrected for elevation difference) reported by the National Weather Service.

8.3 LABORATORY ANALYTICAL EQUIPMENT

The laboratory instruments will be calibrated as specified by the appropriate method before analyzing the test samples. The laboratory instrument calibration procedures are based on instructions in the referenced analytical methods and are summarized, along with other routine quality control checks, in

Table 8-2. The calibrations performed and the results will be reported as appropriate to assure the quality of data in the laboratory sample analysis report.

9.0 ANALYTICAL PROCEDURES

Analytical procedures and methods are summarized in Table 9-1. Individual sampling and analytical methods are described in detail in Attachments A and B of the PDT plan and are incorporated here by reference. Tables 9-2 and 9-3 represent the volatile and semivolatile products of incomplete combustion (PICs) which will be targeted during the analysis of the test samples. These lists represent the target compound list (TCL) to be used by the laboratory. In addition to the TCL compounds, non-TCL peaks greater than 10% of the nearest internal standard will be tentatively identified using a full database library search as described in section 11.3.2 of this plan for all SW-846 Method 8260 and 8270 analyses. Table 9-4 lists the target PAH analytes for CARB Method 429. Table 9-5 lists the target PCB analytes for EPA Method 1668. Table 9-6 lists the target dioxin and furan analytes for SW-846 Method 8290. Table 9-7 lists the target metal analytes by SW-846 Methods 6010 or 6020, and 7470. Table 9-8 lists the target organochlorine pesticide analytes for SW-846 Method 8081.

All analyses will be performed by a laboratory qualified in the categories of sample analysis delineated in this section. Laboratory qualifications can be submitted upon request. The following is a list of the analytical reference methods for the procedures presented in Table 9-1:

- Test Methods for Evaluating Solid Waste, SW-846 (SW-846), Third Edition, November 1986 and Updates
- Sampling and Analysis Methods for Hazardous Waste Incineration, EPA 600/8-84-002.
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series
- Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.
- Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020.
- Performance Specification 4B, Appendix B, 40 CFR 60.

10.0 SPECIFIC INTERNAL QUALITY CONTROL CHECKS

10.1 DEFINITIONS

The various types of QA/QC checks that may be performed as part of the test, both for sampling and analysis, are defined below. One or more of these QA/QC checks are associated with each measurement system in order to assess the compliance of the data to the DQOs established in Section 5.0. Table 10-1 is a summary of all the sample analyses and their associated internal quality control checks associated with this test program.

Audit Sample An audit sample is a field or alternate laboratory prepared blank spike submitted to the test laboratory to assess accuracy or potential sample degradation.

Batch A group of 20 or fewer samples which are prepared together and subjected to the same analytical procedure.

Blank, Field A field blank is a sampling train or sampling component that is set-up in the field but is not used for test sampling. The field blank is used to assess background contamination that may affect the representativeness of the field samples.

Blank, Media A sample of unused sampling media analyzed to ensure the media are uncontaminated. This type of sample may also be referred to as a “reagent blank” (see below).

Blank, Method A method blank is a sample of unused media that is prepared and analyzed in the test laboratory to assess background contamination that may exist in the laboratory, on glassware, or in the analytical system.

Blank, Reagent A sample of unused reagent(s) used to demonstrate the absence of contamination in the reagents.

Blank, Spike A blank spike is a laboratory prepared sample of blank media that is spiked with a known amount of target analyte(s) used to assess the accuracy of the analytical method.

Blank, System An aliquot of uncontaminated reagent used to clean out the analytical system after high level samples have been analyzed or before analysis begins.

Blank, Trip A trip blank is an unused sample component that is shipped to the field along with the sampling equipment/media and/or returned to the laboratory without having been exposed to field conditions. If contamination is encountered in the field blank(s), the trip blank is analyzed to assess whether or not the contamination originates in the field, is inherent in the equipment/media, or results from exposure during shipping and handling.

Breakthrough Check The result of the analysis of a secondary component (i.e., sorbent tube) in a sampling train is compared to the result of the primary component to assess whether or not the primary component has successfully captured the target analytes. If the result of the secondary component analysis is high compared to the primary component analysis, the possibility exists that the analytical results may be artificially low.

Calibration Check A standard solution from a source other than the calibration standards used to verify the integrity of an instrument's calibration.

Calibration Standards High purity compounds or mixtures of compounds used to adjust the response of an analytical instrument. The laboratory will use traceable standards and submit standard preparation logs as part of the deliverables package.

Contingency Sample An archived portion of a field sample from the same location as other field samples that is collected and held in case of breakage or QA/QC failure during the handling or analysis of the primary sample. This type of sample is sometimes referred to as an "archive sample."

Continuing Calibration Verification A mid-point standard, from the same Calibration source as the initial calibration solution analyzed periodically to verify that calibration conditions have not drifted from the initial calibration.

Duplicate Analysis A duplicate is a sample that is split in the laboratory and prepared and analyzed twice. The results of the two analyses are compared as a measure of precision.

Duplicate Injection A second analysis of a single sample preparation. This QC test may be used to assess analytical QC failures, matrix interferences, or as a measure of analytical system precision.

Initial Calibration A series of analyses of solutions, that have known concentrations, used to establish the correspondence between the amount of an analyte present in the solution and the instrument's response across the expected analytical range of the samples. Initial calibrations also establish retention time windows for identification purposes in chromatographic methods.

Interference Check An interference check sample is analyzed, for ICP analysis only, to assess the possible error in analytical results arising from the interaction of various metals in the sample under the conditions of analysis.

Internal Standard Recovery Internal standards are non-target spikes added to samples for quantitation purposes. The percent recovery of the internal standards is checked to assess whether or not significant matrix interferences may affect the accuracy and precision of analytical results.

Performance Evaluation (PE) Sample See Audit Sample.

Proficiency Test A series of blank spikes analyzed in the test laboratory to demonstrate an analyst's ability to successfully perform the method with acceptable precision and accuracy.

Replicate One of a series of identical samples or splits of a single sample used to assess precision.

Serial Dilution The result of the analysis of a highly contaminated sample, run undiluted, is compared to the results for the same sample after serial dilution. The two results are expected to match to within method specified criteria. This test is a measure of the linearity of ICP calibration and the analysis technique.

Spike, Field See Audit Sample.

Spike, Matrix Spike of the known or controlled amount of an actual target analyte to an actual sample matrix that is then analyzed for that analyte. The percent recovery of the spiked analyte provides a measure of the matrix bias.

Surrogates Non-target or isotopically labeled analytes spiked into field samples as a measure of method efficiency and accuracy.

10.2 SPECIFIC QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

A variety of QC checks are required both in the field and in the laboratory to ensure the collection of samples that accurately represent the field conditions under study, to assess compliance with the Data Quality Objectives (DQOs), and to assess biases in the measurement system.

10.2.1 Field Activities

In order to ensure the representativeness of samples collected during the test, and to ensure integrity of field measurements, a variety of QC checks and controls will be implemented throughout the sampling program. These checks and controls will include:

- Standard forms and/or standard field notebooks will be used to document field activities and for data collection. The data collection forms and field notebooks will be reviewed routinely by senior staff for accuracy, completeness, and internal consistency.
- The strict adherence to detailed operating procedures as documented in the various project controlling documents and related SOPs will be enforced by experienced senior technical staff.
- Project personnel will be selected based on appropriate levels of training and experience and will receive project specific training prior to working on-site. Training will include health and safety requirements; security requirements; briefings on overall project goals, objectives, and schedules; and, specific technical training related to their assigned tasks. Training will be documented in the project files.
- Routine calibration will be performed on measurement systems and sampling equipment including metering systems, thermocouples, barometers, rotameters, and pitot tubes. Guidance related to equipment calibration is provided in Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods and Quality Assurance/Quality Control Procedures for Hazardous Waste Incinerators, Appendix A. The detailed specifications, acceptance criteria, and corrective action requirements are presented in Section 8.0 of this QAPP. All calibrations will be documented and the documentation maintained in the project files.
- Leak checks will be performed according to method specifications before and after sampling.
- Field QC samples will be routinely submitted including audit (PE) samples, field blanks, media blanks, reagent blanks, trip blanks, and contingency samples. The frequency of submittal for these field QC samples and other field samples are provided in Tables 5-1 and 8-2.
- Field audits/surveillance will be performed periodically by the QAO to assess conformance to specifications. If nonconforming conditions are noted, the corrective action provisions of the QA plan will be invoked.

10.2.2 Laboratory Activities

Standard laboratory QA procedures, required of each laboratory, provide discussions related to QA/QC checks and controls within the laboratory. Specific data quality objectives, calibration requirements, acceptance criteria, and corrective action requirements for this test program are presented in Table 5-1 and Table 8-2 of this plan.

In addition to the requirements referenced above the laboratory will provide for quality control of sampling media and sample collection equipment. Sorbents used in the organic sampling trains will be prepared according to method specifications. Samples of the prepared media will be tested according to the intended method of use and analysis prior to shipping media to the field. The results of these tests will be retained in the laboratory's files for future reference.

11.0 DATA REDUCTION, DATA VALIDATION, AND DATA REPORTING

11.1 DATA REDUCTION

11.1.1 General Principles

11.1.1.1 Field

Data reduction will occur for the field measurements at the point of sampling. At the point of sampling, the data as measured by the field instrument will be reported in the field notebooks and/or on any forms required for the project.

11.1.1.2 Office

After the field event, the data may be further reduced to data tables, trend analysis tables, or graphs. At any point where manual transcriptions of data take place, an editing function will be invoked to ensure accuracy of the transcriptions.

Upon the return of the analytical results from the laboratory and after data validation, the data will be further reduced to data tables. The data tables will contain the following information:

- Information identifying exactly the samples represented on the tables (e.g. sample location, matrix, etc.),
- The compounds for which the samples were tested,
- The results for each compound, and
- The data flags as applied by the laboratory or by data validators (if used).

11.1.1.3 Laboratory

Data reduction in the laboratory is covered in the Laboratory's QA Manual and SOPs. The laboratory's data reduction process will include at a minimum the following.

- Transcription of data results from raw data printouts to data report forms. This will include any calculations required to report the data in the required units.
- Transcription of QA/QC data onto summary forms to provide the required information for evaluation of the validity of the data. The requirement for each type of data is included in section 11.3.

11.1.2 Specific Data Reduction Requirements

11.1.2.1 GC and GC/MS Techniques

Organic analyses will all be conducted using gas chromatography (GC) techniques. The VOC, SVOC, PAH, OCP, PCB and PCDD/PCDF analyses will employ mass spectral (MS) detectors. Although the principles of operation and specific methods of calibration differ according to the analyte specific methods, the general data reduction scheme is the same for all of these tests. The individual methods should be consulted for details. A summary of the data reduction scheme is presented below.

Depending on the specific method, analytical instrumentation is calibrated at 3 to 5 points covering the expected analytical range. The gas chromatograph/flame ionization detector (GC/FID) or gas chromatograph/electron capture detector (GC/ECD) methods generally employ an external standard calibration technique while the GC/MS methods employ an internal standard technique. For GC/FID and GC/ECD methods, a calibration factor (CF) is calculated using the following formula:

$$CF = \frac{R}{M}$$

Where: CF = Calibration Factor
R = Response or Area of the GC Peak
M = Mass Injected (in nanograms)

The calibration factors must agree to within method specified criteria for the percent relative standard deviation (%RSD). The formula for %RSD is given below.

$$\%\,\mathrm{RSD} = \frac{\sigma}{\mathrm{avg\,CF}} \times 100$$

Where: $\%RSD$ = Percent Relative Standard Deviation
© = Standard Deviation of the Calibration Factors
$avg\ CF$ = Average Calibration Factor

For GC/MS calibrations a response factor (RF) is used rather than the CF. The formula for the RF is:

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}$$

Where: RF = Response Factor
A_s = Response for the Analyte
A_{is} = Response for the Internal Standard
C_s = Concentration of the Analyte
C_{is} = Concentration of the Internal Standard

The RFs must also pass a test of the %RSD in order for the calibration to be considered valid.

When samples are analyzed, the area of the peak produced by a given analyte is compared to the CF or RF to arrive at an analytical result according to the following formula:

$$M_x = A_x \times CF$$

Where:
M_x = The Mass of Analyte in the Sample
A_x = The Response of the Analyte in the Sample
CF = The Calibration (or Response) Factor

Samples containing more of an analyte than the instrument is calibrated for, will have a dilution performed, if such a dilution is practical given the sample preparation method. In that case M_x is multiplied by the dilution factor to arrive at the final result. If, under the circumstances of the method, a dilution is not possible, the analytical result must be considered estimated.

To arrive at a concentration in the gas sample the mass of any sub-samples must be added together and then compared to the volume of gas sampled according to the following formula:

$$C_x = \frac{(M_1 + M_2 + \ldots M_n)}{V}$$

Where: C_x = The Concentration of the Analyte in the Gas Sample
M_n = The Result (Mass) for Each Component in the Sampling Train
V = The Volume of Gas Sampled

11.1.2.2 Analysis of Metals by ICP and Atomic Absorption

The analysis of metals also begins with an instrument calibration at 2 to 6 points, depending on the specific analytical method. For inductively coupled argon plasma spectroscopy (ICP) for non-mercury (non-Hg) metals analyses and cold vapor atomic adsorption spectroscopy (CVAA) for mercury (Hg) analyses, instruments are profiled and calibrated according to the instrument manufacturer's instructions. A calibration blank and a QC check standard are then analyzed to ensure appropriate instrument response. A percent recovery (%R) is calculated according to the following formula:

$$\%R = \frac{Found}{True} \times 100$$

Where: $\%R$ = Percent Recovery
$Found$ = The Result of the Analysis
$True$ = The Expected Result

The %R is expected to be within method specifications before analysis can begin. The calibration is verified periodically according to method specifications using the same technique.

Atomic absorption instruments are calibrated at three to five points. A linear regression is performed and a correlation coefficient is calculated to assess linearity of the curve. It is beyond the scope of this document to provide a detailed explanation of the statistics supporting linear regression and the calculation of the correlation coefficient. Reference can be made to any standard statistics text for additional information. Calibration checks are performed as above and periodically verified.

Analytical results for metals are read directly from the instrument in terms of concentration. Dilution factors must be used as discussed above if applicable. In order to combine the results of the sub-samples of a metals sampling train, the concentration is converted back to mass using the following formula:

$$M_{xs} = C_{xs} \times V_{xs}$$

Where: M_{xs} = Mass in the Sub-sample
C_{xs} = Concentration in the Sub-sample
V_{xs} = Volume of the Sub-sample

The concentration in the gas sample can then be calculated.

11.1.2.3 Ion Chromatography

Anions, such as chloride, are separated on the ion chromatograph using a system comprising separator columns, guard columns, and eluents. The system is calibrated at a minimum of 3 points and the calibration is verified with a mid-range standard. Samples are quantitated in the same manner as given above.

11.1.2.4 Direct Reading Instruments

Gravimetric, temperature, pressure, flow, and CEMS data are directly read from the measurement instrumentation. The instrumentation will be calibration checked prior to the test, and routinely prior to reading measurements, however, no data reduction beyond formatting into tables is expected

11.2 DATA VALIDATION

The results of all sample analysis and all QA/QC sample analysis (100% of the laboratory data) will be compared, step by step, by the QAO or his/her designee, to the specifications given in Tables 5-1 and 8-2. The data validation criteria outlined in: Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis, (1994) and Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis, (1994); prepared by USEPA Data Review Work Group will be followed as applicable to the individual methods used. Any sample data associated with a QC check that fail to meet the target criteria established in these tables will be flagged in the final report, and an assessment of the impact, if any, of missing the target data quality objective will be provided. Additional guidance will be found in the analytical methods and EPA/625/6-89/023, Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration.

Each laboratory providing analysis will be required to provide the results based on the method detection limit (MDL) and either derived reliable detection limits (RDLs) or practical quantitation limits (PQLs) for all non-isotope dilution method compounds. Non-detects for the isotope dilution methods will be determined using the method specific SW-846 definition of a estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory. Results reported between the MDL or EDL and the RDL or PQL will be flagged as estimated. The laboratory must provide include with each data package the basis for and any calculations or statistical methods employed in determining the detection limits used (MDL, EDL and PQL).

Particular attention will be paid to the results of blank data. Analytical data will not be routinely corrected for contamination. They are however evaluated on a case-by-case basis for possible blank correction. A "B" flag will be applied to the samples associated with contaminated blanks such that this information may be assessed in the final report. Risk data will be evaluated against all associated blanks to determine if sample results are less than five times (ten times for common laboratory contaminants) the concentration reported in the blanks. Sample results reported at concentrations less than 5X/10X the blank concentration will be considered not present in the sample and will be qualified as not detected (ND or U).

The output from the data validation process will be a summary comparison of the QA/QC results to the specified data quality objectives, a review and discussion of any deficiencies identified in the data assessments of laboratory performance, and, overall precision and accuracy, representativeness and completeness of the data set.

Detailed procedures for the internal review of data in the laboratory are found in the laboratories QA Manuals and related standard operating procedures (SOPs).

11.3 DATA REPORTING

11.3.1 Experimental Data

Experimental data that will be reported as part of the final test report will include:

- All relevant field measurements in raw and tabular form. This will include, but not necessarily be limited to, calibration data for field instruments, velocity and gas flow measurements, and temperature and pressure measurements.
- Process monitoring data
- All CEMS data to include CO, O2, and THC
- Analytical laboratory data for all laboratory measurements.

The laboratory deliverable package is expected to include the following elements:

The following forms for all organics analyses using Gas Chromatography/Mass Spectroscopy methods:

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Organic analysis data sheet for samples, blanks, and QC analysis (CLP Form 1 or equivalent)
- System monitoring compound/surrogate recoveries summary (CLP Form 2 or equivalent)
- Duplicate analysis summary (CLP Form 3 or equivalent if MS/MSD)

- QC Check Sample summary
- Method blank summary (CLP Form 4 or equivalent) and results
- Instrument performance check summary – tuning reports (CLP Form 5 or equivalent)
- Initial calibration summary (CLP Form 6 or equivalent)
- Continuing calibration check (CLP Form 7 or equivalent)
- Internal standard area and RT summary (CLP Form 8 or equivalent)
- Internal standard recovery summary (PCDD/PCDFs, PAHs and PCBs)
- DDT/Endrin breakdown standards
- Raw data: run logs, mass spectra, quantitation reports, manual integration, and chromatograms for samples, tunes, calibrations, and QC samples, sample preparation logs, and run logs.
- Standards preparation logs and certificate is required
- Sample receipt information including temperature and pH information if preservation is required
- Documentation of all nonconformances and the actions taken
- Examples of all calculations performed
- Detection limits including method detection limits and sample quantitation limits
- Any performance evaluation samples provided
- Percent solids or percent moisture for soil samples

The following forms for all organics analyses using Gas Chromatography:

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Organic analysis data sheet for samples, blanks, and QC samples including retention times of required for the analysis (CLP Form 1 or equivalent)
- System monitoring compound/surrogate recoveries summary (CLP Form 2 or equivalent)
- Duplicate analysis summary (CLP Form 3 or equivalent if MS/MSD)
- QC Check Sample summary
- Method blank summary and results (CLP Form 4 or equivalent)
- Initial calibration summary (CLP Form 6 or equivalent)
- Calibration verification summary (CLP Form 7 or equivalent)
- Graphic Representation of Curve Fit, with Correlation Coefficient
- Analytical Sequence (run logs)
- Raw data: quantitation reports, manual integrations, and chromatograms, and retention times for each column in all field and QC samples, sample preparation logs, and run logs.

- Standards preparation logs and certificates (if applicable)
- Sample receipt information including temperature and pH information if preservation is required
- Documentation of all nonconformances and the actions taken
- Examples of all calculations performed
- Compound confirmation (if required for the analysis)
- Peak Resolution Summary (if required for analysis)
- Retention time window determination
- Detection limits including method detection limits and sample quantitation limits
- Any performance evaluation samples provided
- Percent solids or percent moisture for soil samples

The following forms for all metals analyses:

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references (SOPs if necessary)
- Inorganic analysis data sheet (CLP Form 1 or equivalent)
- Initial and continuing calibration verification (CLP Form 2 or equivalent)
- Blanks summary (CLP Form 3 or equivalent)
- Spike sample recovery/Post digest spike sample recovery (CLP Form 5 or equivalent)
- Interference checks sample results (CLP Form 4 or equivalent)
- Serial dilution results (CLP Form 9 or equivalent)
- Duplicate results (CLP Form 6 or equivalent)
- Laboratory control sample results (CLP Form 7 or equivalent)
- Raw data for samples, blanks, QC samples, calibrations, and instrument checks, sample preparation logs and, instrument run logs.
- Documentation of all nonconformances and the actions taken
- Sample receipt information including temperature and pH information if preservation is required
- Examples of all calculations performed
- Detection limits including method detection limits, instrument detection limits, and quantitation limits
- Method of standard additions data
- ICP-AES inter-element correction (IEC) factors
- ICP-MS tunes
- ICP-MS internal standards relative intensity summary
- Any performance evaluation samples provided

- Standards preparation logs and certificates (if applicable)
- Percent solids or percent moisture for soil samples
- AA – wavelengths used for analysis

The following forms for all inorganic non-metals analyses: (as appropriate for analysis)

- Case narrative and sample identification cross reference
- Copies of Chain of Custody documentation
- Method summaries and references
- Inorganic analysis data sheet
- Calibration summaries
- Method blank results summary
- Sample spike recovery
- Duplicate sample results
- Laboratory Control Sample.
- Raw data, chromatograms, area printouts, sample preparation logs and, instrument run logs
- Examples of all calculations performed
- Documentation of all nonconformances and the actions taken
- Sample receipt information including temperature and pH information if preservation is required
- Standards preparation logs and certificates (if applicable)
- Percent solids or percent moisture for soil samples
- Logbook pages (gravimetric)
- Scale calibrations (gravimetric)

11.3.2 Reporting of Tentatively Identified Compounds

In addition to the target analytes identified for volatile and semivolatile organic stack gas analysis, there are generally a number of non-target components observed in the chromatogram. Attempts to identify and quantify these unknown chromatographic peaks can improve the percentage of identified organic compounds and reduce overall uncertainty. However, because the instrument is not calibrated for these unknown compounds, the identification and quantitative analysis is tentative until the identification is confirmed by the analysis of a standard. EPA OSW risk assessment guidance recommends that TICs be considered “identified” compounds for purposes of site-specific risk assessments to ensure that appropriate credit is given to defensible efforts to identify the maximum number of organic compounds.

To identify non-target TICs, the mass spectrum can be searched against a computerized library of reference mass spectra. A forward library search selects the largest mass spectral peaks from the unknown mass spectrum and looks for reference spectra in the library that contain the peaks of the unknown. A reverse library search looks for the peaks in the reference spectrum that occur in the unknown mass spectrum. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library matches should the analyst assign a tentative identification. Any components that are identified are referred to as TICs, since no reference standard was analyzed at the same time as the unknown. Without calibration of the instrument with the actual compound, TICs are quantified using the nearest-eluting internal standard with a relative instrument response factor of 1.00. The resulting concentration is considered “estimated,” because the response factor is not compound-specific. An unknown level of error in the quantitation is introduced using the response factor of 1.00; this level of error will vary from compound to compound.

Methods 8260/8270 present guidelines for identification of TICs, and these guidelines are summarized below:

- Relative intensities of major ions in the reference mass spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample mass spectrum.
- The relative intensities of the major ions should agree within ±20 percent. Example: for an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance should lie between 30 and 70 percent.
- Molecular ions present in the reference mass spectrum should be present in the sample mass spectrum.
- Ions present in the sample mass spectrum but not in the reference mass spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference mass spectrum but not in the sample mass spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library programs can sometimes create these discrepancies.

If, in the judgment of the experienced mass spectral interpreter, no valid tentative identification can be made, the compound should be reported as “unknown.” The mass spectral interpreter should give additional classification of the unknown compound, if possible (i.e., unknown aromatic compound, unknown hydrocarbon, unknown chlorinated compound). If a probable molecular weight can be distinguished, this molecular weight should also be reported. The experienced interpreter should apply this experience and judgment to the mass spectral interpretations supplied by the computerized library search. For example, if a hydrocarbon occurring 40 minutes into the chromatographic analysis is identified by the computer as “octane,” analytical judgment dictates that this identification is scientifically illogical and the compound should be reported as “unknown hydrocarbon.” By no means should the computer identifications be accepted uncritically.

11.3.3 Project Reporting Format

The format for the PDT Report final report is outlined in Figure 11-1.

11.3.4 Detection Limits and Data Reduction

Detection limits for organic compounds will be derived as recommended by the U.S. EPA Office of Solid Waste (OSW) in the “Human Health Risk Assessment Protocol” published in July 1998. This protocol recommends that non-isotope dilution methods quantify non-detects using the method detection limit (MDL), derived reliable detection limits (RDL). The MDL is defined in 40 CFR Part 136 Appendix A. Each laboratory will be required to provide MDLs for each non-isotope dilution target compound along with the RDLs. Isotope dilution methods quantify non-detects using estimated detection limits (EDLs) as defined in SW-846 Method 8290.

Detection limits for inorganic compound compliance data will be derived according to the referenced analytical method and the laboratory's standard operating procedures. Each laboratory will be required to provide detection limits for each inorganic analyte.

Analytical results that are reported as “Not Detected” (ND) or “Below the detection Limit” (BDL) in compliance demonstration samples will be handled in the following manner. The analytical result will be reported as “ND”, “BDL”, or “U” and the appropriate detection limit as discussed above will be shown. The detection limit will be used as the assumed sample concentration in all subsequent calculations. For analyses of single component samples (e.g., feed or residue samples) that are reported as “ND” or “BDL”, the results of all subsequent calculations using those values will be accompanied by a “less than” (“<”) sign. For analyses of multi-component samples (e.g., VOST, Method 0010), where the analytical results must be combined for use in subsequent calculations, the detection limit will be used for each component reported as “ND” or “BDL”. Results of calculations utilizing values derived from multi-component analyses where the analyte of concern was non-detect in one or more components but not in all of the components, the result will be accompanied by a “<” sign. If the analyte of concern in a multi-component sample is non- detect in all the components of a particular sample, the result will be shown with a “<” sign, and will also be marked as “ND” or “BDL”. Where destruction and removal efficiency or similar performance measurements are calculated using emissions rates reported with a “<” value, the resulting performance measurement will be accompanied by a “greater than” (“>”) sign.

For the purpose of risk assessment, if all results for a compound are found to be nondetect, one half of the detection limit will be use.

11.3.5 Final Case Files

At a minimum, the following documents will be retained upon the completion of the project in the final case file, which must be maintained at the WCAI facility for a period at least five years:

- All legal documents and orders,
- All field documents including those used for preliminary field activities,
- Copies of all analytical data,
- Copies of the final report and background documents, and
- All correspondence relating to the project as well as corrective action requests.

12.0 ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES

12.1 SAMPLING EQUIPMENT

All equipment used in emission testing measuring systems must be maintained in good operating order. To achieve this objective, a routine preventive maintenance program is necessary. Procedures used in this program follow those outlined in Maintenance Calibration and Operation of Isokinetic Source Sampling Equipment, Publication No. APTD-05-76 and Volume III of the Quality Assurance Handbook for Air Pollution Measurement Systems.

The potential impact of equipment malfunction on data completeness is minimized through two complementary approaches. First, an equipment maintenance program is part of routine operations. The maintenance program's strengths include:

- Trained technicians experienced in the details of equipment maintenance and fabrication,
- Adequate spare parts inventory, and
- The availability of tools and specialized equipment.

The second approach is based upon equipment redundancy. Backup equipment, spare parts and tools are included on the materials transported to the field for each sampling task. This approach allows the sampling team to respond to equipment breakage or malfunction in a timely fashion, minimizing the quantity of lost data.

For field equipment, preventive maintenance schedules are based on the results of routine inspections and on accumulated experience. At a minimum, equipment will be inspected prior to the beginning of and at the conclusion of each test. A record of each inspection (Figure 12-1) will be kept as part of the final case file. Maintenance schedules for continuous emissions monitors follow manufacturer's recommendations.

Each item of field test equipment is assigned a unique, permanent identification number. An effective preventive maintenance program is necessary to ensure data quality. Each item of equipment returning from the field is inspected before it is returned to storage. During the course of these inspections, items are cleaned, repaired, reconditioned and recalibrated where necessary. Each item of equipment transported to the field for this test program is inspected again before being packed to detect equipment problems that may originate during periods of storage. This minimizes lost time on the job site due to equipment failure. Occasional equipment failure in the field is unavoidable despite the most rigorous inspection and maintenance procedures. For this reason, adequate spare parts are kept in a central location so the sampling contractor can quickly respond to the job site with replacement equipment for all critical sampling train components.

12.2 LABORATORY INSTRUMENTS

The laboratories perform regular maintenance on all analytical instruments. An inventory of replacement parts is kept to prevent downtime. Manufacturers' service representatives are also contracted, as required, for major instrument repairs.

Preventive and routine maintenance is covered in each of the laboratories' QA Manuals and SOPs or in accordance with manufacturer's recommendations (i.e., instrument manuals). Daily maintenance (such as replacement of injector septa, etc.) is covered in instrument SOPs. Inoperative equipment is tagged as non-usable until repairs are performed. Logbooks are maintained for each instrument to record usage, maintenance, and repairs.

12.3 PROCESS INSTRUMENTS

On-site personnel perform regular maintenance on all process instrumentation. Routine and preventive maintenance procedures are documented and updated as required. Where appropriate, manufacturers' recommendations for maintenance of process instruments are followed. Operators conduct daily reviews of process instrumentation by noting suspicious or inconsistent readings. Maintenance logs are used to record the frequency and type of repairs necessary for process instruments. Process instruments used to demonstrate compliance with operating limits will be calibrated prior to the test. Records of these calibrations will be included in the final test report.

13.0 ASSESSMENT PROCEDURES FOR ACCURACY, PRECISION, & COMPLETENESS

The QA activities implemented in this study will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 5.0 discusses the QA activities that will generate the accuracy and precision data for each sample type. The generalized forms of the equations that will be used to calculate accuracy and precision are presented below.

13.1 ACCURACY

When a reference standard material is used in the analysis, percent Accuracy (A) will be calculated as follows:

$$A = \frac{\text{Found concentration}}{\text{True concentration}} \times 100$$

Percent analyte Recovery (R) will be calculated as follows:

$$R = \frac{X - N}{S} \times 100$$

Where X is the experimentally determined value, N is the amount of native material in the sample, and S is the amount of spiked material of the species being measured. Recoveries are used to determine accuracy when standards are not available, or are not appropriate for a given matrix.

13.2 PRECISION

When less than three analyses of the same parameter are available, precision will be calculated as a Relative Percent Difference (RPD) from the average of replicate measurements according to:

$$\text{RPD} = \frac{(X_1 - X_2)}{\text{Average } X} \times 100$$

Where X_1 and X_2 are the highest and lowest results of replicate measurements.

Where three or more analyses of the same parameter are available, the precision will be determined as the Relative Standard Deviation (RSD) according to:

$$\mathrm{RSD} = \frac{\text{Standard deviation}}{\text{Average X}} \times 100$$

13.3 COMPLETENESS

Completeness of data generated from a test program is usually calculated as follows:

$$\%\ \text{Completeness} = \frac{\text{Valid data}}{\text{Expected data}} \times 100$$

Data completeness is defined in Section 5.0 of this QAPP as the percentage of valid data collected from the total number of valid tests conducted. Three valid test runs, at each test condition, are required for the test to be completed. If an individual sample from a test run is lost or broken, the data for that individual analytical parameter may not be 100% complete. This, however, may not invalidate the test run. The completeness objective for this test program is to generate sufficient data for the regulatory agencies to judge the performance of the system.

14.0 AUDIT PROCEDURES, CORRECTIVE ACTION, AND QA REPORTING

14.1 PERFORMANCE AND SYSTEM AUDITS

This section presents information related to the procedures used by the QA staff to assess conformance of the project staff to the specifications contained in the relevant project controlling documents. Further, auditing may be employed to assess the ability of subcontractors to successfully perform the work.

14.1.1 Field Audits

The QAO assigned to the project will conduct audits of the operations at the site to ensure that work is being performed in accordance with the various project controlling documents and associated standard operating procedures. A checklist appropriate to the activities scheduled during the audit will be used. The audit will cover, but not necessarily be limited to, such areas as:

- Conformance to SOPs
- Completeness and accuracy of documentation
- Chain of custody procedures
- Compliance with Health and Safety requirements.

These audits may occur at the start, during, or end of each significant phase of the project.

14.1.2 Performance Evaluations

The QAO and/or the Regulatory Agencies may submit Performance Evaluation (PE) samples (referred to elsewhere as "Audit Samples") to the laboratory as indicated on Tables 5-1 and 6-2. PE samples may be submitted for analysis to demonstrate analytical performance on an as required basis.

14.1.3 Office Audits

The QAO will also conduct periodic audits of the case files. These audits will assess the completeness of the files and verify that all of the appropriate information is included in the files.

14.1.4 Laboratory Audits

WCAI or its appointed representative may choose to audit the laboratories at any time during the course of the project on an as-required basis to assess the laboratory's ability to successfully perform the work and to ensure mutual agreement between WCAI and the laboratory with regard to the scope of work, QA/QC requirements, and deliverable requirements. Reasonable notice will be provided prior to any on-site inspection of the laboratory.

14.2 CORRECTIVE ACTION

The following procedures have been established to ensure that nonconforming conditions, such as malfunctions, deficiencies, deviations and errors are promptly investigated, documented, evaluated and corrected. Every person employed in the test is expected to function as a QC inspector to ensure the quality of the final product. Quality, as it relates to this project, is defined as "performing the work according to the agreed upon specifications contained in the PDT plan and relevant SOPs or causing the specification to be changed *in a controlled manner*." Each individual is encouraged to identify any condition adverse to the successful completion of the work or any modification to the specifications that might result in a better end product. These improvements might be framed in terms of higher quality, greater safety, greater efficiency, and/or lower cost. However, it can not be stressed strongly enough, that only documented and approved changes to the specifications are allowable.

14.2.1 Field

When a nonconforming condition or an opportunity for improvement is noted at the site or contractor location, the corrective action provisions of this plan will be invoked to identify the condition and recommend corrective action. Condition identification, cause, reference documents and the corrective action planned to be taken will be documented and reported at a minimum to the employee's immediate supervisor.

A Corrective Action Request (CAR), as shown in Figure 14-1, should be used to identify the adverse condition or opportunity for improvement, reference document(s) and recommended corrective action(s). The CAR is directed to the Test Coordinator. The Test Coordinator affixes his signature and the date to the corrective action block that states the cause of the condition(s) and corrective action(s) to be taken. The Test Coordinator is responsible for first notifying the regulatory agency representative of any problems or deviations from the QAPP, or PDT plan identified in the CAR. The Test Coordinator then forwards the requested response to the QAO for follow-up and filing. The QAO maintains the log for status control of CARs and responses confirms the adequacy of the intended corrective action(s) and verifies its implementation. The QAO will issue and distribute copies of completed CARs to the originator, Test Coordinator, WCAI Test Project Manager, and the involved contractor(s) if any. CARs are transmitted to the project file for future reference, and are incorporated into the final test report.

An audit checklist may be used to assist in identifying items which may require corrective actions. An example audit checklist, which can be adapted for use in multiple situations, is shown as Figure 14-2.

Testing activities may be impacted by a number of factors, including process interruptions, operating conditions which are outside of specifications, inclement weather, or sampling train difficulties. A set of field troubleshooting guidelines has been developed and presented in Table 14-1 to assist in recognizing and resolving these issues in the field.

14.2.2 Laboratory

The laboratories' QA Manuals and the related SOPs, contain detailed discussions of corrective actions to be taken if established criteria fail during laboratory analysis. The laboratory has the responsibility to immediately notify the Test Coordinator and/or QAO when any analytical QC nonconformance occurs, so a mutually acceptable course of action can be pursued.

14.3 QA REPORTS TO MANAGEMENT

The QAO will provide a written report to the Test Coordinator. This report will address:

- Overview of activities and significant events related to QA/QC
- Summary of audit results
- Review of corrective action request status
- Laboratory QA/QC reports
- Data validation reports
- Summary of significant changes in procedures or QA/QC programs
- Recommendations.

Upon project completion, a Final QA Report will be issued, assessing the overall degree of project conformance to specifications and the impact of any nonconforming conditions on data quality that may affect management decisions. This report will be incorporated into the final test report.

The nature of the laboratories' Quality Assurance reports is provided in their respective Laboratory Quality Assurance Manuals and SOPs. Where no other specifications exists, the laboratory must conform to the provisions given in this section.

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Spent Activated Carbon	Chloride & Elemental analysis	90 – 110% of reference value for analysis of a known material conducted once per test series for each property of concern	<10% RPD for duplicate analyses conducted for one sample from each matrix. (viscosity – every sample in duplicate)	An optional matrix spike/matrix spike duplicate may be conducted if desired. If conducted, the desired spiking level is 2 times the native concentration or 10 times the detection limit, whichever is greater. Accuracy and precision objectives are as stated for reference material and duplicate analyses.
Spent Activated Carbon, Makeup Water, Scrubber Blowdown, POTW Discharge & Caustic	Volatile organics	Matrix Spike % recoveries as specified in Table 5-2 Surrogate % recoveries (Table 5-2) spiked onto every field sample	< 35% RPD for duplicate preparation and analysis conducted for one sample from each matrix <35% PRD for matrix spike/matrix spike duplicate analysis	Analysis of one method blank per sample batch carried through all preparation and analysis steps. Results should be less than the lowest calibration standard.
Spent Activated Carbon, Makeup Water, Scrubber blowdown, POTW Discharge & Caustic	Semivolatile organics	Matrix Spike % recoveries as specified in Table 5-2 Surrogate % recoveries (Table 5-2) spiked onto every field sample	< 35% RPD for duplicate analyses conducted for one sample from each matrix. < 35% RPD for duplicate analysis of spiked sample . (Matrix Spike Duplicate).	Analysis of one method blank per sample batch, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Spent Activated Carbon, Makeup Water, Scrubber blowdown, POTW Discharge & Caustic	Metals	70 - 130% recovery of each metal of concern spiked into an aliquot of one sample from each matrix. The spiking level should be the greater of either 1 - 2 times the apparent concentration in the unspiked sample, or at least 10 times the detection limits. (Matrix Spike).	< 35% RPD for duplicate analyses conducted for one sample from each matrix. This criterion only applies to individual metals with an apparent concentration greater than the lowest calibration standard used in the analyses. and/or < 35% RPD for duplicate analysis of the spiked sample. (Matrix Spike Duplicate).	Analysis of one method blank per sample batch, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	Volatile Organics (Method 0030, VOST)	75 - 125% recovery of standards (independent of calibration standards) spiked onto 2 VOST tube pairs and analyzed prior to sample analysis. Surrogate % recoveries as specified in Table 5-2 spiked onto every field sample (VOST tubes and condensate). 50 - 150% of true value for analysis of samples collected from EPA audit cylinder (if requested and provided).	< 25% RPD between spike recoveries from 2 VOST tube pairs analyzed prior to sample analysis. < 35% RSD of surrogate spike recoveries between each field sample.	Separate analysis of front and back tubes from each pair should show less than 30% of the front tube concentration on the back tube. This criterion is not applicable for a particular compound if the back tube contains less than 75 ng of that compound. One pair of field blank tubes is analyzed for every test run (6 samples). Should be less than the lowest calibration standard. One pair of trip blank tubes, accompanying each tube shipment from the field, should be analyzed if the field blanks show contamination. Should be less than the lowest calibration standard. One pair of laboratory blank tubes (prepared in the same batch as the field sample tubes, and archived) should be analyzed if trip blanks show contamination. Should be less than the lowest standard. System blanks are analyzed daily before sample analysis, and between high-level samples. Should be less than the lowest standard.

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	PCDD/PCDFs (Method 0023A)	70 - 130% recovery of isotopically labeled PCDD/PCDF pre-sampling surrogates spiked onto each sorbent resin tube prior to sampling. 40 - 130% recovery of isotopically labeled tetra- through hexa-chlorinated PCDD/PCDF internal surrogate standards spiked onto train components prior to extraction. 25 - 130% recovery of isotopically labeled hepta- and octa- chlorinated PCDD/PCDF internal surrogate standards spiked onto train components prior to extraction. 50 – 150% recovery of isotopically labeled alternate standards spiked onto the extract prior to cleanup standards and recovery standards spiked onto the extract following cleanup. 50 - 150% of true value for analysis of an audit sample (if requested/provided)	< 30% RSD of spike recoveries between samples for labeled compounds spiked prior to sampling. < 35% RSD of spike recoveries between samples for internal quantitation standards	Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train is performed to assess contamination. Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be conducted to assess contamination.

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	Hexavalent chromium (Method 0061)	60 - 140% recovery of Cr^{+6} spiked into one sample preparation. The spiking level should be the greater of either 1 - 2 times the apparent concentration in the unspiked sample, or at least 10 times the detection limits. 90 - 110% of the true value for analysis of an audit sample obtained from the regulatory agency (if requested).	< 30% RPD for duplicate analysis of every sample including the spiked sample.	Analysis of one set of reagent blanks, carried through all preparation and analysis steps, should be less than 5% of sample levels or below the detection limit.
Stack gas	Semivolatile organics (Method 0010)	Recovery of isotopically labeled POHCs or appropriate surrogates spiked into each sample as listed in Table 5-2 Recovery of POHCs or appropriate surrogates spiked onto a **blank XAD-2 resin trap** and one condensate sample in the laboratory as specified in Table 5-2. (Matrix Spike)	< 40% RPD (or < 35% RSD, if greater than 2 determinations are made) of spike recoveries between field samples. < 35% RPD for duplicate injection from one run. This criterion is not applicable if the compound is found at a concentration below the lowest calibration standard. < 35% RPD for duplicate preparation and analysis of spiked blank XAD-2 resin trap. (Matrix Spike Duplicate)	Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit. Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	PCBs (Method 0010) (Draft EPA Method 1668A)	Recovery of isotopically labeled PCBs or appropriate surrogates spiked into each train component prior to extraction as specified in Table 5-2. Recovery of PCBs spiked onto **a blank XAD-2 resin trap** and one condensate sample in the laboratory as specified in Table 5-2. (Matrix Spike)	< 40% RPD (or < 35% RSD, if greater than 2 determinations are made) of spike recoveries between field samples. < 35% RPD for duplicate injection from one run. This criterion is not applicable if the particular compound is found at a concentration below the lowest calibration standard. < 350% RPD for duplicate preparation and analysis of spiked blank XAD-2 resin trap. (Matrix Spike Duplicate)	Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit. Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.
Stack gas	PAHs (Method 0010) (CARB Method 429)	50 - 150% recovery of isotopicaly labeled PAH compounds spiked onto each sorbent resin tube prior to sampling 50 - 150% recovery of isotopicaly labeled internal quantitation standards spiked onto train components prior to extraction 50 - 150% recovery of PCBs spiked onto **a blank XAD-2 resin trap** and one condensate sample in the laboratory. (Matrix Spike)	< 30% RSD of spike recoveries between samples for labeled compounds spiked prior to sampling. < 35% RSD of spike recoveries between samples for internal quantitation standards	Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train is performed to assess contamination Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be conducted to assess contamination

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	OCP (Method 0010) (SW846 Method 8081)	Recovery of appropriate surrogates spiked into each sample as listed in Table 5-2 Recovery of appropriate surrogates spiked onto a **blank XAD-2 resin trap** and one condensate sample in the laboratory as specified in Table 5-2. (Matrix Spike)	< 40% RPD (or < 35% RSD, if greater than 2 determinations are made) of spike recoveries between field samples. < 35% RPD for duplicate injection from one run. This criterion is not applicable if the compound is found at a concentration below the lowest calibration standard. < 35% RPD for duplicate preparation and analysis of spiked blank XAD-2 resin trap. (Matrix Spike Duplicate)	Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit. Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.
Stack gas	Total semivolatile and nonvolatile organics (Method 0010)	TCO – Daily QC sample $\pm$ 15% of actual value GRAV – Audit sample $\pm$ 20% of actual value	TCO - <15% RPD between analysis of one replicate sample per test GRAV - <20% RPD between duplicate analysis of each sample.	Analysis of one method blank for recovery reagents and XAD/filter carried through all preparation and analysis steps should be conducted to assess contamination. Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train is performed to assess contamination

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	Total volatile organics (Method 0040)	Tedlar Bag - 80 - 120% of the true value for analysis of a gas with known hydrocarbon concentration placed into a clean bag and analyzed prior to sample analysis.	< 35% RPD for duplicate analysis of known gas	Analyze one gas bag filled with zero air or zero nitrogen, and one water blank with each field sample should be less than 20% of the sample levels or below the detection limit. Results are evaluated on a case-by-case basis for possible blank correction.
Stack Gas	Metals (Method 29)	75 - 125% recoveries for each metal of concern spiked into an aliquot of one sample preparation from each train component. The spiking level should be the greater of either 1 - 2 times the apparent concentration in the unspiked sample, or at least 10 times the detection limits. 70 - 125 % recoveries for spiked blank filters for each metal of concern. 75 - 125% recoveries for preparation and analysis of two complete blank trains spiked with each metal of concern. The spiking level should be the greater of either 1 - 2 times the expected concentration in the field samples, or at least 10 times the detection limit.	< 20% RPD between analyses of the two blank spiked filters for each metal of concern. < 20% RPD between analyses of the two spiked blank trains. For mercury only, < 25% RPD for duplicate analysis conducted for every sample.	Analysis of one set of reagent blanks, carried through all preparation and analysis steps. Results are evaluated on a case-by-case basis for possible blank correction.
Stack Gas	Particulate (Method 26A)	99 – 101% agreement for balance calibration check with Class-S weights prior to and after all tare weighing and after all tare weighing and gravimetric determinations.	Duplicate weighing of each sample must be within 0.5 mg or 1% total tare weight, whichever is greater.	None

Table 5-1. Test Analytical Data Quality Objectives

Sample Matrix	Test Parameters	Accuracy Objectives	Precision Objectives	Other Objectives
Stack gas	HCl/Cl_2 (Method 26A)	85 – 115% recovery of chloride spiked into an aliquot of both acidified and alkaline impinger solutions at less than 3 times the apparent concentration in the unspiked sample or 10 times the detection limit, whichever is greater. (Matrix Spike)	<25% RPD for duplicate analysis conducted for each acidified and each alkaline impinger solution sample. This criterion is relaxed to <50% RPD if the sample concentration is less than 5 times the detection limit. <25% RPD for duplicate analysis of each spiked sample. (MS/MSD)	Analysis of one method blank analyzed in duplicate per sample batch, carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.
Stack gas	Particle Size distribution (PSD) via Cascade Impactor (CARB Method 501)	N/A	N/A	Analysis of one set of blank filter media to establish analysis baseline.
Stack gas	Carbon monoxide (permanent CEMS)	Daily calibration checks (high and low range) within 3% of span.	40 CFR 63 Subpart EEE Appendix; PA Continuous Source Monitoring Manual	N/A
Stack gas	Oxygen (permanent CEMS)	Daily calibration checks within 0.5% oxygen.	40 CFR 63 Subpart EEE Appendix; PA Continuous Source Monitoring Manual	N/A
Stack gas	THC/VOC (Temporary CEMS)	Pre- and Post-run calibration checks within 3% of respective span calibration gases	40 CFR 63 Subpart EEE Appendix; PA Continuous Source Monitoring Manual; EPA Method 25A	N/A

$$\text{Relative Percent Difference (RPD)} = \frac{\text{highest value} - \text{lowest value}}{\text{average value}} \times 100$$

$$\%\ \text{Recovery} = \frac{\text{found - native}}{\text{amount spiked}} \times 100$$

$$\%\ \text{Accuracy} = \frac{\text{found concentration}}{\text{actual concentration}} \times 100\ \text{(for reference materials)}$$

$$\text{Relative Standard Deviation} = \frac{\text{standard deviation}}{\text{average value}} \times 100$$

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Spent activated carbon feed, Makeup Water, Scrubber Blowdown, POTW Discharge & Caustic	Volatile Organics in Organic & Aqueous Liquid Matrices and Solid Matracies		
	Surrogate Spikes	Toluene-d_8	50 – 130%
		4-Bromofluorobenzene	50 – 130%
		1,2-Dichloroethane-d_4	50 – 130%
	Matrix Spikes	Chlorobenzene	50 – 130%
		Tetrachloroethene	50 – 130%
		1,1-Dichloroethene	50 – 130%
		Trichloroethene	50 – 130%
		Benzene	50 – 130%
		Toluene	50 – 130%
	Internal Standards (area count compared to Continuing calibration)	Fluorobenzene	50 – 200%
		Chlorobenzene-d_5	50 – 200%
		1,4-dichlorobenzene-d_4	50 – 200%
Makeup Water, Scrubber blowdown, POTW Discharge & Caustic	Semivolatile Organics in Organic & Aqueous Liquid Matrices		
		Nitrobenzene-d_5	23 - 120%
	Surrogate Spikes	2-Fluorobiphenyl	30 - 115%
		Phenol-d_5	24 - 113%
		2-Fluorophenol	25 - 121%
		2,4,6-Tribromophenol	19 - 122%
		1,4-Dichlorobenzene-d_4	50 – 200%
	Internal Standards (area count compared to Continuing calibration)	Naphthalene-d_8	50 – 200%
		Acenaphthene-d_{10}	50 – 200%
		Phenanthrene-d_{10}	50 – 200%
		Chrysene-d_{12}	50 – 200%
		Perylene-d_{12}	50 – 200%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Spent activated carbon feed	Semi-volatile Organics Solid Matrices		
	Surrogate Spikes (to sample fraction before extraction)	Nitrobenzene-d_5	23 - 120%
		2-Fluorobiphenyl	30 – 115%
		Phenol-d_5	24 – 113%
		2-Fluorophenol	25 – 121%
		2,4,6-Tribromophenol	19 – 122%
	Matrix Spikes	Phenol	25 – 150%
		2-Chlorophenol	26 - 150%
		1,4-Dinitrophenol	28 - 150%
		N-Nitroso-di-n-propylamine	41 - 150%
		1,2,4-Trichlorobenzene	38 - 150%
		4-Chloro-3-methylphenol	26 - 150%
		Acenaphthene	31 - 150%
		4-Nitrophenol	11 - 150%
		2,4-Dinitrotoluene	28 - 150%
		Pentachlorotoluene	17 - 150%
		Pyrene	35 - 150%
	Internal Standards (area count compared to Continuing calibration)	1,4-Dichlorobenzene-d_4	50 – 200%
		Naphthalene-d_8	50 – 200%
		Acenaphthene-d_{10}	50 – 200%
		Phenanthrene-d_{10}	50 – 200%
		Chrysene-d_{12}	50 – 200%
		Perylene-d_{12}	50 – 200%
Stack Gas Volatile Organics (Method 0030)	VOST Solid Matrices		
	Surrogate Spikes	Toluene-d_8	50 – 130%
		4-Bromofluorobenzene	50 – 130%
		1,2-Dichloroethane-d_4	50 – 130%
	Internal Standards (area count compared to Continuing calibration)	Fluorobenzene	50 – 200%
		Chlorobenzene-d_5	50 – 200%
		1,4-dichlorobenzene-d_4	50 – 200%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas Volatile Organics (Method 0030)	VOST Condensate (Aqueous Matrices)		
	Surrogate Spikes	Toluene-d_8	50 – 130%
		4-Bromofluorobenzene	50 – 130%
		1,2-Dichloroethane-d_4	50 – 130%
	Matrix Spikes (or Method Spike)	1,1-Dichloroethene	50 – 130%
		Trichloroethene	50 – 130%
		Benzene	50 – 130%
		Toluene	50 – 130%
		Chlorobenzene	50 – 130%
	Internal Standards (area count compared to Continuing calibration)	Fluorobenzene	50 – 200%
		Chlorobenzene-d_5	50 – 200%
		1,4-dichlorobenzene-d_4	50 – 200%
Stack Gas PCDD/PCDFs (Method 0023A)	PCDD/PCDFs Solid and Aqueous Matracies		
	Pre-sampling Surrogate Spikes (to XAD-2 resin before field use and to the filter immediately prior to extraction)	$^{37}Cl_4$-2,3,7,8-TCDD	70 – 130%
		$^{13}C_{12}$-2,3,4,7,8-PeCDF	70 – 130%
		$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	70 – 130%
		$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	70 – 130%
		$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	70 – 130%
	Internal Surrogate Spikes (to sample fraction before extraction)	$^{13}C_{12}$-2,3,7,8-TCDF	40 – 130%
		$^{13}C_{12}$-2,3,7,8-TCDD	40 – 130%
		$^{13}C_{12}$-1,2,3,7,8-PeCDF	40 – 130%
		$^{13}C_{12}$-1,2,3,7,8-PeCDD	40 – 130%
		$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	40 – 130%
		$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	40 – 130%
		$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	25 – 130%
		$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	25 – 130%
		$^{13}C_{12}$-OCDD	25 – 130%
	Alternate Standard (to extract before cleanup)	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	50 – 150%
	Recovery Standards (to extract before analysis)	$^{13}C_{12}$-1,2,3,4-TCDD	50 – 150%
		$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	50 – 150%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas Semivolatile Organics (Method 0010)	Semi-volatile Organics Solid Matrices (Filter and XAD-2 Resin)		
	Sampling Surrogate (to XAD-2 resin before field use)	$^{13}C_3$-labeled Naphthalene	50 - 150%
	Surrogate Spikes (to sample fraction before extraction)	Nitrobenzene-d_5	23 - 120%
		2-Fluorobiphenyl	30 – 115%
		Phenol-d_5	24 – 113%
		2-Fluorophenol	25 – 121%
		2,4,6-Tribromophenol	19 – 122%
	Matrix Spikes (Blank Spiked Resin)	Phenol	25 – 150%
		2-Chlorophenol	26 - 150%
		1,4-Dinitrophenol	28 - 150%
		N-Nitroso-di-n-propylamine	41 - 150%
		1,2,4-Trichlorobenzene	38 - 150%
		4-Chloro-3-methylphenol	26 - 150%
		Acenaphthene	31 - 150%
		4-Nitrophenol	11 - 150%
		2,4-Dinitrotoluene	28 - 150%
		Pentachlorotoluene	17 - 150%
		Pyrene	35 - 150%
	Internal Standards (area count compared to Continuing calibration)	1,4-Dichlorobenzene-d_4	50 – 200%
		Naphthalene-d_8	50 – 200%
		Acenaphthene-d_{10}	50 – 200%
		Phenanthrene-d_{10}	50 – 200%
		Chrysene-d_{12}	50 – 200%
		Perylene-d_{12}	50 – 200%
Stack Gas Semivolatile Organics (Method 0010)	Semi-volatile Organics Aqueous Matrices (Condensate)		
	Surrogate Spikes (to sample fraction before extraction)	Nitrobenzene-d_5	35 – 114%
		2-Fluorobiphenyl	43 – 116%
		Phenol-d_5	10 – 94%
		2-Fluorophenol	21 – 100%
		2,4,6-Tribromophenol	10 – 123%
	Matrix Spikes or Method Spike)	Phenol	12 – 150%
		2-Chlorophenol	27 – 150%
		1,4-Dinitrophenol	36 – 150%
		N-Nitroso-di-n-propylamine	41 – 150%
		1,2,4-Trichlorobenzene	39 – 150%
		4-Chloro-3-methylphenol	23 – 150%
		Acenaphthene	46 – 150%
		4-Nitrophenol	10 – 150%
		2,4-Dinitrotoluene	24 – 150%
		Pentachlorotoluene	9 – 150%
		Pyrene	26 – 150%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas PCBs (Method 0010)	PCBs Solid or Aqueous Matrices (Filter and XAD-2 Resin, or Condensate)		
	Sampling Surrogate Spikes * (to XAD-2 resin before field use)	$^{13}C_{12}$-2,4,4'-Tri-CB $^{13}C_{12}$-2,3,3',5,5'-PeCB $^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	60 - 140% 60 - 140% 60 - 140%
	Surrogate Spikes (to sample fraction before extraction)	$^{13}C_{12}$-3,3',4,4'-TCB $^{13}C_{12}$-2,3,3',4,4'-PeCB $^{13}C_{12}$-2,3',4,4',5-PeCB $^{13}C_{12}$-3,3',4,4',5-PeCB $^{13}C_{12}$-2,3,3',4,4',5-HxCB $^{13}C_{12}$-2,3,3',4,4',5'HxCB $^{13}C_{12}$-2,3',4,4',5,5'-HxCB $^{13}C_{12}$-3,3',4,4',5,5'-HxCB $^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB $^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB $^{13}C_{12}$-DCB [a]	24 – 169% 21 – 178% 21 – 178% 21 – 178% 26 – 152% 26 – 152% 26 – 152% 26 – 152% 23 – 143% 23 – 143% 26 - 152%
	Cleanup Standard Spikes (to extract before cleanup)	$^{13}C_{12}$-3,4,4',5-TCB $^{13}C_{12}$-2,3,4',5,5'-PeCB	35 – 197% 35 – 197%
	Internal Standard Spikes * (to extract after cleanup and before analysis)	$^{13}C_{12}$-2,2',5,5'-TCB $^{13}C_{12}$-2,2',4,5,5'-PeCB $^{13}C_{12}$-2,2',3,4,4',5'-HxCB $^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	40 – 120% 40 – 120% 40 – 120% 40 – 120%
	Matrix Spike * (blank spiked resin)	3,3'4,4'-TCB 2,3,3',4,4'-PeCB 2,3,4,4',5-PeCB 2,3,4,4',5-PeCB 2',3,4,4',5-PeCB 3,3',4,4',5-PeCB 2,3,3'4,4'5-HxCB 2,3,3',4,4',5'-HxCB 2,3',4,4',5,5'-HxCB 3,3',4,4'5,5'-HxCB 2,2'3,3',4,4',5-HpCB 2,2'3,4,4',5,5'-HpCB 2,3,3',4,4'5,5'-HpCB	60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140% 60 - 140%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas OCPs (Method 0010)	Organochlorinie Pestidicides Solid Matrices (Filter and XAD-2 Resin)		
	Surrogate Spikes (to sample fraction before extraction)	Decachlorobiphenyl	50 – 150%
		Tetrachloro-m-xylene	50 – 150%
	Matrix Spikes (Blank Spiked Resin)	Aldrin	70 – 130%
		α-BHC	70 – 130%
		β-BHC	70 – 130%
		γ-BHC	70 – 130%
		δ-BHC	70 – 130%
		Chlorobenzilate	70 – 130%
		α-Chlordane	70 – 130%
		γ-Chlordane	70 – 130%
		DBCP	70 – 130%
		4,4'-DDD	70 – 130%
		4,4'-DDE	70 – 130%
		4,4'-DDT	70 – 130%
		Diallate	70 – 130%
		Dieldrin	70 – 130%
		Endosulfan I	70 – 130%
		Endosulfan II	70 – 130%
		Endosulfan sulfate	70 – 130%
		Endrin	70 – 130%
		Endrin aldehyde	70 – 130%
		Endrin ketone	70 – 130%
		Heptachlor	70 – 130%
		Heptachlor epoxide	70 – 130%
		Isodrin	70 – 130%
		Methoxychlor	70 – 130%
		Toxaphene	70 – 130%
			70 – 130%
	Internal Standards (optional) (area count compared to Continuing calibration)	Pentachloronitrobenzene	50 – 200%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas OCP (Method 0010)	Organochlorine Pesticides Aqueous Matrices (Condensate)		
	Surrogate Spikes (to sample fraction before extraction)	Decachlorobiphenyl	50 – 150%
		Tetrachloro-m-xylene	50 – 150%
	Matrix Spikes (or Method Spike)	Aldrin	70 – 130%
		α-BHC	70 – 130%
		β-BHC	70 – 130%
		γ-BHC	70 – 130%
		δ-BHC	70 – 130%
		Chlorobenzilate	70 – 130%
		α-Chlordane	70 – 130%
		γ-Chlordane	70 – 130%
		DBCP	70 – 130%
		4,4'-DDD	70 – 130%
		4,4'-DDE	70 – 130%
		4,4'-DDT	70 – 130%
		Diallate	70 – 130%
		Dieldrin	70 – 130%
		Endosulfan I	70 – 130%
		Endosulfan II	70 – 130%
		Endosulfan sulfate	70 – 130%
		Endrin	70 – 130%
		Endrin aldehyde	70 – 130%
		Endrin ketone	70 – 130%
		Heptachlor	70 – 130%
		Heptachlor epoxide	70 – 130%
		Isodrin	70 – 130%
		Methoxychlor	70 – 130%
		Toxaphene	70 – 130%
			70 – 130%
	Internal Standards (optional) (area count compared to Continuing calibration)	Pentachloronitrobenzene	50 – 200%

Table 5-2. Organic Surrogate Spike and Matrix Spike Recovery Limits

Sample Matrix	QA Parameter	Spiking Compound	Recovery Limits
Stack Gas PAHs (Method 0010)	PAH Solid or Aqueous Matrices (Filter and XAD-2 Resin, or Condensate)		
	Sampling Surrogate (to XAD-2 resin before field use)	d_{10}-Fluorene	50 – 150%
		d_{14}-Terphenyl	50 – 150%
	Internal Standard Spikes (to sample fraction before extraction)	Naphthalene-d_8	50 – 150%
		2-Methylnaphthalene	50 – 150%
		Acenaphthene-d_{10}	50 – 150%
		Phenanthrene-d_{10}	50 – 150%
		Fluoranthene-d_{10}	50 – 150%
		Benzo(a)anthracene-d_{12}	50 – 150%
		Chrysene-d_{12}	50 – 150%
		Perylene-d_{12}	50 – 150%
		Benzo(b)fluoranthene-d_{12}	50 – 150%
		Benzo(d)fluoranthene-d_{12}	50 – 150%
		Benzo(a)pyrene-d_{12}	50 – 150%
		Benzo(g,h,I)perylene-d_{12}	50 – 150%
		Indeno(1,2,3-c,d)pyrene-d_{12}	50 – 150%
		Dibenzo(a,h)anthracene-d_{14}	50 – 150%
	Alternate Standard (add before extraction)	Anthracene-d_{10}	50 – 150%
	Recovery Standards (after extraction and before GC/MS)	Acenaphthene-d_{10}	50 – 150%
		Pyrene-d_{10}	50 – 150%
		Benxo(e)pyrene-d_{12}	50 – 150%
	Matrix Spike (blank spiked resin)	Naphthalene	50 – 150%
		2-Methylnaphthalene	50 – 150%
		Acenaphthalene	50 – 150%
		Acenaphthylene	50 – 150%
		Fluorene	50 – 150%
		Phenanthrene	50 – 150%
		Anthracene	50 – 150%
		Fluoranthene	50 – 150%
		Pyrene	50 – 150%
		Benzo(a)anthracene	50 – 150%
		Chrycene	50 – 150%
		Benzo(b)fluoranthene	50 – 150%
		Benzo(k)fluoranthene	50 – 150%
		Benzo(e)pyrene	50 – 150%
		Benzo(a)pyrene	50 – 150%
		Perylene	50 – 150%
		Indeno(1,2,3-cd)pyrene	50 – 150%
		Dibenz(a,h)anthracene	50 – 150%
		Benzo(ghi)perylene	50 – 150%

* CARB 428 limits. None provided in EPA Method 1668A
a No limits provided for this surrogate. The most stringent limits from the other surrogates were used.

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
VOLATILE ORGANICS					
Acetone	67-64-1	2.00E-01	9.44E-02	3.33E+00	6.10E-06
Acrylonitrile	107-13-1	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Benzene	71-43-2	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Bromochloromethane	74-97-5	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Bromodichloromethane	75-27-4	4.00E-02	1.89E-02	6.67E-01	1.22E-06
Bromoform	75-25-2	6.00E-02	2.83E-02	1.00E+00	1.83E-06
Bromomethane	74-83-9	5.00E-02	2.36E-02	8.33E-01	1.53E-06
2-Butanone (MEK)	78-93-3	3.00E-01	1.42E-01	5.00E+00	9.15E-06
Carbon Disulfide	75-15-0	3.00E-02	1.42E-02	5.00E-01	9.15E-07
Carbon Tetrachloride	56-23-5	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Chlorobenzene	108-90-7	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Chlorodibromomethane	124-48-1	6.00E-02	2.83E-02	1.00E+00	1.83E-06
Chloroethane	75-00-3	4.00E-02	1.89E-02	6.67E-01	1.22E-06
Chloroform	67-66-3	3.00E-02	1.42E-02	5.00E-01	9.15E-07
Chloromethane	74-87-3	1.00E+00	4.72E-01	1.67E+01	3.05E-05
1,2-Dibromoethane (a)	106-93-4	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Dibromomethane	74-95-3	5.00E-02	2.36E-02	8.33E-01	1.53E-06
Dichlorodifluoromethane	75-71-8	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1-Dichloroethane	75-34-3	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,2-Dichloroethane	107-06-2	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1-Dichloroethene	75-35-4	4.00E-02	1.89E-02	6.67E-01	1.22E-06
1,2-Dichloropropane	78-87-5	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Dicyclopentadiene (a)	77-73-6	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Ethylbenzene	100-41-4	3.00E-02	1.42E-02	5.00E-01	9.15E-07
2-Ethyl-1-methylbenzene (a)	611-14-3	9.00E-01	4.25E-01	1.50E+01	2.75E-05
2-Hexanone	591-78-6	2.00E-01	9.44E-02	3.33E+00	6.10E-06
Iodomethane	74-88-4	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Methyl methacrylate (a)	80-62-6	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Methylene Chloride	75-09-2	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Propylbenzene (a)	103-65-1	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Styrene	100-42-5	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1,2,2-Tetrachloroethane	79-34-5	3.00E-02	1.42E-02	5.00E-01	9.15E-07
Tetrachloroethene	127-18-4	6.00E-02	2.83E-02	1.00E+00	1.83E-06
Tetrahydrofuran (a)	109-99-9	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Triethylamine (a)	121-44-8	9.00E-01	4.25E-01	1.50E+01	2.75E-05
Toluene	108-88-3	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1,1-Trichloroethane	71-55-6	5.00E-02	2.36E-02	8.33E-01	1.53E-06
1,1,2-Trichloroethane	79-00-5	8.00E-02	3.78E-02	1.33E+00	2.44E-06
Trichloroethene	79-01-6	1.00E+00	4.72E-01	1.67E+01	3.05E-05
Trichlorofluoromethane	75-69-4	1.00E-01	4.72E-02	1.67E+00	3.05E-06
1,2,3-Trichloropropane	96-18-4	2.00E-02	9.44E-03	3.33E-01	6.10E-07
1,2,4-trimethylbenzene (a)	95-63-6	9.00E-01	4.25E-01	1.50E+01	2.75E-05
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	2.00E-01	9.44E-02	3.33E+00	6.10E-06
Vinyl Acetate	108-05-4	1.00E-01	4.72E-02	1.67E+00	3.05E-06
Vinyl Chloride	75-69-4	1.00E-03	4.72E-04	1.67E-02	3.05E-08
m & p-Xylenes	108-38-3/106-42-3	2.00E-03	9.44E-04	3.33E-02	6.10E-08
o-Xylene	95-47-6	2.00E-03	9.44E-04	3.33E-02	6.10E-08
Xylenes (total)	1330-02-7	2.00E-03	9.44E-04	3.33E-02	6.10E-08

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
SEMIVOLATILE ORGANICS					
Aniline	62-53-3	1.50E+01	1.42E-01	5.00E+00	9.15E-06
Benzoic Acid (a)	65-85-0	2.00E+01	1.89E-01	6.66E+00	1.22E-05
Benzyl Alcohol	100-51-6	4.00E+01	3.77E-01	1.33E+01	2.44E-05
Bis(2-chloroethoxy) Methane	111-91-1	2.00E+00	1.89E-02	6.66E-01	1.22E-06
Bis-(2-chloroethyl) Ether	111-44-4	2.00E+00	1.89E-02	6.66E-01	1.22E-06
Bis(2-ethylhexyl) Phthalate	117-81-7	5.00E+00	4.72E-02	1.67E+00	3.05E-06
4-Bromophenyl-phenyl Ether	101-55-3	2.00E+00	1.89E-02	6.66E-01	1.22E-06
Butylbenzylphthalate	85-68-7	3.00E+00	2.83E-02	9.99E-01	1.83E-06
4-Chloroaniline	106-47-8	1.00E+01	9.43E-02	3.33E+00	6.10E-06
4-Chloro-3-methylphenol	59-50-7	4.10E+00	3.87E-02	1.37E+00	2.50E-06
2-Chloronaphthalene	91-58-7	1.50E+00	1.42E-02	5.00E-01	9.15E-07
2-Chlorophenol	95-57-8	2.20E+00	2.08E-02	7.33E-01	1.34E-06
4-Chlorophenyl-phenyl Ether	7005-72-3	2.20E+00	2.08E-02	7.33E-01	1.34E-06
Dibenzofuran	132-64-9	2.20E+00	2.08E-02	7.33E-01	1.34E-06
Di-n-butylphthalate	84-74-2	1.30E+00	1.23E-02	4.33E-01	7.93E-07
1,2-Dichlorobenzene	95-50-1	2.00E+00	1.89E-02	6.66E-01	1.22E-06
1,3-Dichlorobenzene	541-73-1	2.30E+00	2.17E-02	7.66E-01	1.40E-06
1,4-Dichlorobenzene	106-46-7	2.40E+00	2.26E-02	7.99E-01	1.46E-06
3,3'-Dichlorobenzidine	91-94-1	1.30E+01	1.23E-01	4.33E+00	7.93E-06
2,4-Dichlorophenol	120-83-2	3.00E+00	2.83E-02	9.99E-01	1.83E-06
Diethyl Phthalate	84-66-2	2.80E+00	2.64E-02	9.33E-01	1.71E-06
2,4-Dimethylphenol	105-67-9	9.80E+00	9.25E-02	3.26E+00	5.98E-06
Dimethylphthalate	131-11-3	1.60E+00	1.51E-02	5.33E-01	9.76E-07
1,3-Dinitrobenzene	99-65-0	1.00E+01	9.43E-02	3.33E+00	6.10E-06
4,6-Dinitro-2-methylphenol	534-52-1	1.40E+01	1.32E-01	4.66E+00	8.54E-06
2,4-Dinitrophenol	51-28-5	2.90E+01	2.74E-01	9.66E+00	1.77E-05
2,4-Dinitrotoluene	121-14-2	3.10E+00	2.92E-02	1.03E+00	1.89E-06
2,6-Dinitrotoluene	606-20-2	2.60E+00	2.45E-02	8.66E-01	1.59E-06
Di-n-octyl Phthalate	117-84-0	5.00E+00	4.72E-02	1.67E+00	3.05E-06
1,4-Dioxane	123-91-1	1.00E+01	9.43E-02	3.33E+00	6.10E-06
Diphenylamine	122-39-7	3.00E+00	2.83E-02	9.99E-01	1.83E-06
Hexachlorobenzene	118-74-1	2.10E+00	1.98E-02	7.00E-01	1.28E-06
Hexachlorobutadiene	87-68-3	2.90E+00	2.74E-02	9.66E-01	1.77E-06
Hexachlorocyclo-pentadiene	77-47-4	2.30E+01	2.17E-01	7.66E+00	1.40E-05
Hexachloroethane	67-72-1	3.80E+00	3.58E-02	1.27E+00	2.32E-06
Isophrone	78-59-1	5.00E+00	4.72E-02	1.67E+00	3.05E-06
2-Methylphenol	95-48-7	6.10E+00	5.75E-02	2.03E+00	3.72E-06
3/4-Methylphenol	106-44-5	5.20E+00	4.91E-02	1.73E+00	3.17E-06
2-Nitroaniline	88-74-4	8.00E+00	7.55E-02	2.66E+00	4.88E-06
3-Nitroaniline	99-09-2	6.00E+00	5.66E-02	2.00E+00	3.66E-06
4-Nitroaniline	100-01-6	3.00E+00	2.83E-02	9.99E-01	1.83E-06
Nitrobenzene	98-95-3	4.70E+00	4.43E-02	1.57E+00	2.87E-06
2-Nitrophenol	88-75-5	8.10E+00	7.64E-02	2.70E+00	4.94E-06
4-Nitrophenol	100-02-7	1.00E+01	9.43E-02	3.33E+00	6.10E-06
N-Nitrosodimethylamine	62-44-2	5.00E+00	4.72E-02	1.67E+00	3.05E-06
N-Nitrosodiphenylamine	86-30-6	5.00E+00	4.72E-02	1.67E+00	3.05E-06
N-Nitroso-di-n-propylamine	621-64-7	2.50E+00	2.36E-02	8.33E-01	1.52E-06
2,2'-oxybis (1-Chloropropane)	108-60-1	1.10E+01	1.04E-01	3.66E+00	6.71E-06
Pentachlorobenzene	608-93-5	1.20E+01	1.13E-01	4.00E+00	7.32E-06
Pentachlorophenol	87-86-5	2.80E+00	2.64E-02	9.33E-01	1.71E-06
Phenol	108-95-2	1.00E+01	9.43E-02	3.33E+00	6.10E-06
1,2,4-Trichlorobenzene	120-82-1	4.40E+00	4.15E-02	1.47E+00	2.68E-06
2,4,5-Trichlorophenol	95-95-4	3.10E+00	2.92E-02	1.03E+00	1.89E-06
2,4,6-Trichlorophenol	88-06-2	2.40E+00	2.26E-02	7.99E-01	1.46E-06

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
POLYAROMATIC HYDROCARBONS					
Acenaphthene	83-32-9	8.00E-03	7.55E-05	2.66E-03	4.88E-09
Acenaphthylene	208-96-8	8.10E-03	7.64E-05	2.70E-03	4.94E-09
Anthracene	120-12-7	9.70E-03	9.15E-05	3.23E-03	5.91E-09
Benzo(a)anthracene	56-55-3	8.80E-03	8.30E-05	2.93E-03	5.37E-09
Benzo(b)fluoranthene	205-99-2	9.90E-03	9.34E-05	3.30E-03	6.04E-09
Benzo(k)fluoranthene	207-08-9	9.80E-03	9.25E-05	3.26E-03	5.98E-09
Benzo(g,h,i)perylene	191-24-2	6.90E-03	6.51E-05	2.30E-03	4.21E-09
Benzo(a)pyrene	50-32-8	7.80E-03	7.36E-05	2.60E-03	4.76E-09
Benzo(e)pyrene	192-97-2	7.80E-03	7.36E-05	2.60E-03	4.76E-09
Chrysene	218-01-9	1.00E-02	9.43E-05	3.33E-03	6.10E-09
Dibenzo(a,h)anthracene	53-70-3	1.20E-02	1.13E-04	4.00E-03	7.32E-09
Fluoranthene	206-44-0	9.20E-03	8.68E-05	3.06E-03	5.61E-09
Fluorene	86-73-7	8.50E-03	8.02E-05	2.83E-03	5.18E-09
Indeno(1,2,3-cd)pyrene	193-39-5	1.20E-02	1.13E-04	4.00E-03	7.32E-09
2-Methylnaphthalene	91-57-6	2.00E-02	1.89E-04	6.66E-03	1.22E-08
Naphthalene (b)	91-20-3	5.00E-01	4.72E-03	1.67E-01	3.05E-07
Perylene	198-55-0	9.00E-03	8.49E-05	3.00E-03	5.49E-09
Phenanthrene	85-01-8	8.50E-03	8.02E-05	2.83E-03	5.18E-09
Pyrene	129-00-0	7.20E-03	6.79E-05	2.40E-03	4.39E-09
ORGANOCHLORINE PESTICIDES					
1,2-Dibromo-3-chloropropane	96-12-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
4,4'-DDD	72-54-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
4,4'-DDE	72-5-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
4,4'-DDT	50-29-3	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Aldrin	309-00-2	1.00E+00	9.43E-03	3.33E-01	6.10E-07
alpha-BHC	319-84-6	1.00E+00	9.43E-03	3.33E-01	6.10E-07
beta-BHC	319-85-7	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Lindane	58-89-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
gamma-BHC	319-86-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Chlorobenzilate	510-15-6	1.00E+00	9.43E-03	3.33E-01	6.10E-07
alpha-Chlordane	5103-71-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
gamma-Chlordane	5103-74-2	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Diallate	2303-16-4	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endosulfan I	959-98-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endosulfan II	33213-65-9	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endosulfan sulfate	1031-07-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endrin	72-20-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Endrin Ketone	53494-70-5	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Heptachlor	76-44-8	1.00E+00	9.43E-03	3.33E-01	6.10E-07
Heptachlor epoxide	1024-57-3	1.00E+00	9.43E-03	3.33E-01	6.10E-07

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
POLYCHLORINATED BIPHENYLS					
3,4,3',4'-Tetrachlorobiphenyl	32598-13-3	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,4',5-Tetrachlorobiphenyl	70362-50-4	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,3',4'-Pentachlorobiphenyl	32598-14-4	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,5,4'-Pentachlorobiphenyl	74472-37-0	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,4,5,3',4'-Pentachlorobiphenyl	31508-00-6	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,5,2',4'-Pentachlorobiphenyl	65510-44-3	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,5,3',4'-Pentachlorobiphenyl	57465-28-8	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,5,3',4'-Hexachlorobiphenyl	38380-98-4	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,3',4',5'-Hexachlorobiphenyl	68782-90-7	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,4,5,3',4',5'-Hexachlorobiphenyl	52663-72-6	5.00E-04	4.72E-06	1.67E-04	3.05E-10
3,4,5,3',4',5'-Hexachlorobiphenyl	32774-16-6	5.00E-04	4.72E-06	1.67E-04	3.05E-10
2,3,4,5,3',4',5'-Heptachlorobiphenyl	39635-31-9	5.00E-04	4.72E-06	1.67E-04	3.05E-10
DIOXINS AND FURANS					
2,3,7,8-TCDD	1746-01-6	6.00E-06	5.66E-08	2.00E-06	3.66E-12
2,3,7,8-TCDF	51207-31-9	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,7,8-PeCDD	40321-76-4	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,7,8-PeCDF	57117-41-6	5.00E-06	4.72E-08	1.67E-06	3.05E-12
2,3,4,7,8-PeCDF	57117-31-4	5.00E-06	4.72E-08	1.67E-06	3.05E-12
1,2,3,6,7,8-HxCDD	57653-85-7	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,4,7,8-HxCDD	39227-28-6	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,7,8,9-HxCDD	19408-74-3	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,6,7,8-HxCDF	57117-44-9	4.00E-06	3.77E-08	1.33E-06	2.44E-12
1,2,3,4,7,8-HxCDF	70648-26-9	5.00E-06	4.72E-08	1.67E-06	3.05E-12
1,2,3,7,8,9-HxCDF	72918-21-9	6.00E-06	5.66E-08	2.00E-06	3.66E-12
2,3,4,6,7,8-HxCDF	60851-34-5	5.00E-06	4.72E-08	1.67E-06	3.05E-12
1,2,3,4,6,7,8-HpCDD	35822-39-4	6.00E-06	5.66E-08	2.00E-06	3.66E-12
1,2,3,4,6,7,8-HpCDF	67562-39-4	7.00E-06	6.60E-08	2.33E-06	4.27E-12
1,2,3,4,7,8,9-HpCDF	55673-89-7	9.00E-06	8.49E-08	3.00E-06	5.49E-12
Total OCDD	3268-87-9	1.00E-05	9.43E-08	3.33E-06	6.10E-12
Total OCDF	39001-02-0	2.00E-05	1.89E-07	6.66E-06	1.22E-11

Table 5-3. Estimated Stack Gas Detection Limits - Target Analytes

Compound	CAS Number	Estimated Detection Limit (ug/sample)	Estimated Detection Limit (ug/dscf)	Estimated Detection Limit (ug/dscm)	Emission Rate at Estimated Detection Limit (g/s)
METALS					
Aluminum	7429-90-5	8.40E+00	1.40E-01	4.94E+00	9.05E-06
Antimony	7440-36-0	1.20E+00	2.00E-02	7.06E-01	1.29E-06
Arsenic	7440-38-2	6.00E-01	1.00E-02	3.53E-01	6.46E-07
Barium	7440-39-3	7.00E-01	1.17E-02	4.12E-01	7.54E-07
Beryllium	7440-41-7	3.00E-01	5.00E-03	1.77E-01	3.23E-07
Cadmium	7440-43-9	3.00E-01	5.00E-03	1.77E-01	3.23E-07
Chromium (Total)	7440-47-3	5.00E-01	8.33E-03	2.94E-01	5.39E-07
Chromium (Hexavalent)	7440-47-3	1.50E-01	2.50E-03	8.83E-02	1.62E-07
Cobalt	7440-48-4	1.50E+00	2.50E-02	8.83E-01	1.62E-06
Copper	7440-50-8	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Lead	7439-92-1	3.00E+00	5.00E-02	1.77E+00	3.23E-06
Manganese	7439-96-5	9.00E-01	1.50E-02	5.30E-01	9.70E-07
Mercury	7439-97-6	1.60E+00	2.67E-02	9.42E-01	1.72E-06
Nickel	7440-02-0	1.50E+00	2.50E-02	8.83E-01	1.62E-06
Selenium	7782-49-2	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Silver	7440-22-4	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Thallium	7440-28-0	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Vanadium	7440-62-2	1.00E+00	1.67E-02	5.89E-01	1.08E-06
Zinc	7440-66-6	8.00E-01	1.33E-02	4.71E-01	8.62E-07

(a) Compound not on typical target analyte list, and does not have proven sampling and/or analytical performance. Detection limit estimated.
(b) Naphthalene can be analyzed by low resolution GC/MS as a semivolatile compound or by high resolution GC/MS as a PAH compound.

Basis for stack gas flow rate (dscfm)	3878
Assumed VOC sample size (L, dry, std.)	60
Assumed SVOC sample size (dscf)	106
Assumed PAH sample size (dscf)	106
Assumed PCDD/PCDF sample size (dscf)	106
Assumed metals sample size (dscf)	60

Note: All estimates for PCDD/PCDFs and PAHs are estimated EDLs. Other compounds are estimated MDLs.
SVOC values are based on a 2-way split of a dedicated sampling train (analyzed fraction and archive fraction).

Note: All estimates are for the entire sampling train. For example, volatile organics include three sets of VOST tubes plus condensate.

dscf = dry standard cubic feet
dscm = dry standard cubic meters
Standard conditions 68°F, 29.92 in. Hg

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location[a]	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method[b]
1	Spent Activated Carbon (1-Volatiles) (1-Semivolatiles) (1 – Metals) (1 - Properties) (1-Archive)	Conveyor	Teflon scoop 4L glass jug, 250 ml jar (VOA) 1L glass bottles with teflon lined lids	1 scoop per grab; 250 ml volatiles 1L semivolatiles 1L properties 1L metals 1L archive	Collect a grab sample at each 15-minute interval during each test run. Grab samples will be combined in a glass jug to build run composite. Collect four 1-lter samples and one 250 ml VOA jar of the homogenized composite at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.3
2	Makeup water (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at the beginning of the test; Fill 4L bottle at beginning of test. Fill three 1-liter samples from the 4L bottle.	SW-846, Vol. II, Chapter 9, Section 9.2
3	Caustic (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at the beginning of the test; Fill 4L bottle at beginning of test. Fill three 1-liter samples from the 4L bottle.	SW-846, Vol. II, Chapter 9, Section 9.2
4	Scrubber Blowdown (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA ~200 ml per grab; 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at each 30 minute interval; Collect a ~200 ml grab sample at each 30-minute interval during each test run. Grab samples will be combined in a glass jug to build run composite. Collect three 1-liter samples of the homogenized composite at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.2
5	POTW Discharge (2-Volatiles) (1-Semivolatiles) (1 – Metals) (1-Archive)	Tap	40 ml vials; 4L glass jug, 1L glass bottles with teflon lined lids	40 ml VOA ~200 ml per grab; 1L semivolatiles 1L metals 1L archive	Collect one pair of 40 ml VOA vials at each 30 minute interval; Collect a ~200 ml grab sample at each 30-minute interval during each test run. Grab samples will be combined in a glass jug to build run composite. Collect three 1-liter samples of the homogenized composite at the end of the test run.	SW-846, Vol. II, Chapter 9, Section 9.2

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location[a]	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method[b]
Stack (6)	Stack gas M29	Port	EPA Method 29 multiple metals sampling train	Minimum 120 minutes[c,d]	Collect integrated sample for metals and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5, and 29.
Stack (6)	Stack gas M0061	Port	SW-846 Method 0061 hexavalent chromium sampling train	Minimum 120 minutes[c,d]	Collect integrated samples for hexavalent chromium and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0061
Stack (6)	Stack gas M26A	Port	EPA Method 26A sampling train	Minimum 120 minutes[c,d]	Collect integrated sample for particulate, hydrogen chloride, and chlorine. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5, and 26A
Stack (6)	Stack gas M0010-SV	Port	SW-846 Method 0010 sampling train	Minimum 3 dry standard cubic meters[c,d]	Collect integrated sample for semivolatile organics, organochlorine pesticides, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010.
Stack (6)	Stack gas M0010-P	Port	Combined SW-846 Method 0010, EPA CARB Method 429 sampling train	Minimum 3 dry standard cubic meters[c,d]	Collect integrated sample for PAHs, PCBs, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010; CARB Method 429.

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location[a]	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method[b]
Stack (6)	Stack gas M0010-TOE	Port	SW-846 Method 0010 sampling train	Minimum 3 dry standard cubic meters[c,d]	Collect integrated samples for total semivolatile organics, total nonvolatile organics, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0010; EPA TOE Guidance
Stack (6)	Stack gas M0023A	Port	SW-846 Method 0023A sampling train	Minimum 3 hours and 2.5 dry standard cubic meters[c,d]	Collect integrated sample for PCDD/PCDFs, and moisture. Measure stack gas velocity, pressure, and temperature. Collect bag samples or use CEM for oxygen and carbon dioxide.	EPA Methods 1 through 5; SW846-0023A.
Stack (6)	Stack gas M0030	Port	SW-846 Method 0030 volatile organic sampling train	4 tube pairs per run; 40 minutes per tube pair. Up to 20 liters of stack gas per tube pair	Collect four pairs of sorbent tubes and stack gas condensate for volatile organcs during each run.	SW846-0030 (VOST)
Stack (6)	Stack gas M0040	Port	SW-846 Method 0040 sampling train	25 – 50 liters	Collect representative sample through a heated sample probe and filter; through a condenser and into a Tedlar bag. Transport dried sample and condensate to GC/FID.	EPA Methods 1 through 5; SW846-0040; EPA TOE Guidance.
Stack (6)	Stack gas PSD	Port	EPA Method 5 sampling train	As required	Collect particle size distribution samples on filter media	EPA Method 5
Stack (6)	Stack gas CEMS	Port	Temporary CEMS THC	Continuous	Continuously monitor stack gas for total hydrocarbons during each run	EPA Method 25A
Stack (7)	Stack gas CEMS	Port	Installed CEMS CO	Continuous	Continuously monitor stack gas carbon monoxide during each run.	40 CFR 63 Subpart EEE Appendix; PS 4B

Table 6-1 Sample Collection Locations, Equipment, and Methods

Location[a]	Sample Name Number	Access	Equipment	Sample Size	General Procedure/Frequency	Reference Method[b]
Stack (7)	Stack gas CEMS	Port	Installed CEMS O_2	Continuous	Continuously monitor stack gas oxygen during each run.	40 CFR 63 Subpart EEE Appendix; PS 4B

a Refer to Figure 5-1 of the Comprehensive Performance Test Plan.

b "SW846" refers to Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.
"EPA Method" refers to New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60.
"CARB" refers to California Air Resources Board Methods.
"PS 4B" refers to Performance Specification 4B, 40 CFR 60.
"EPA TOE Guidance" refers to Guidance for Total Oganics, EPA/600/R-96/033, March 1996

c The exact volume of gas sampled will depend on the isokinetic sampling rate.

d Isokinetic sampling trains include:
- Collecting one set of bag samples (or using CEM) for oxygen and carbon dioxide analysis to determine stack gas molecular weight (EPA Method 3)
- Performing stack gas velocity, pressure, and temperature profile measurement for each sampling location (EPA Method 2)
- Determining the moisture content of the stack gas for each sampling train (EPA Method 4)

Table 6-2. Summary of Expected Trial Burn Field Samples

Sample Matrix	Container	Routine Samples or Field Splits (a) (No. per Run)	Number of Runs	Total Samples Collected During Test
Spent Activated Carbon				
Physical/chemical properties	1L glass	1	3	3
Volatile organics	250 mL VOA jar	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		5		15
Makeup Water				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
Caustic				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
Scrubber Blowdown				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
POTW Discharge				
Volatile organics	40 ml VOA vials	1	3	3
Semivolatile organics	1L glass	1	3	3
Metals	1L glass	1	3	3
Archive	1L glass	1	3	3
Subtotal		4		12
Stack Gas M0023A				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half acetone/methylene chloride and toluene rinses	500 ml amber glass	1	3	3
Back half acetone/methylene chloride and toluene rinses	500 ml amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half acetone/methylene chloride and toluene rinses (blank train)	500 ml amber glass	NA	NA	1
Back half acetone/methylene chloride and toluene rinses (blank train)	500 ml amber glass	NA	NA	1
XAD-2 spiked resin trap blanks	Glass trap	NA	NA	2
Acetone reagent blank	500 ml amber glass	NA	NA	1
Methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Toluene reagent blank	500 ml amber glass	NA	NA	1
Audit sample (XAD-2 resin trap)	Glass trap	NA	NA	1
Subtotal		4		22
Stack Gas M0010-SV (Modified for SVOCs and OCP)				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Back half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Condensate	1 liter amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
Back half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
XAD-2 Resin trap blanks	Glass trap	NA	NA	2
Acetone reagent blank	500 ml amber glass	NA	NA	1
Methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Subtotal		5		23
Stack Gas M0010-P (Modified for PAH and PCB)				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Back half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Condensate	1 liter amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
Back half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
XAD-2 Resin trap blanks	Glass trap	NA	NA	2
Acetone reagent blank	500 ml amber glass	NA	NA	1
Methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Subtotal		5		23

Table 6-2. Summary of Expected Trial Burn Field Samples

Sample Matrix	Container	Routine Samples or Field Splits (a) (No. per Run)	Number of Runs	Total Samples Collected During Test
Stack Gas VOST M0030				
Tenax resin tube	Glass culture tube	4	3	12
Tenax resin/charcoal tube	Glass culture tube	4	3	12
Condensate	40 ml VOA	1	3	3
Tenax resin tube field blank	Glass culture tube	1	3	3
Tenax resin/charcoal tube field blank	Glass culture tube	1	3	3
Tenax resin tube spiked resin blank	Glass culture tube	NA	NA	1
Tenax resin/charcoal tube spiked resin blank	Glass culture tube	NA	NA	1
Tenax resin tube trip blank (1 per shipment to lab)	Glass culture tube	NA	NA	1
Tenax resin/charcoal tube trip blank (1 per shipment to lab)	Glass culture tube	NA	NA	1
Tenax resin tube audit samples (4 tubes x 1 cylinder)	Glass culture tube	4	NA	4
Tenax resin/charcoal tube audit samples (4 tubes x 1 cyl)	Glass culture tube	4	NA	4
Subtotal		19		45
Stack Gas Metals Method 29				
Filter	Glass petri dish	1	3	3
Nitric acid probe rinse	500 ml amber glass	1	3	3
Acidified peroxide impinger solution and rinses	1 liter amber glass	1	3	3
Initially empty impinger solution and rinse	500 ml amber glass	1	3	3
Permanganate impinger solution and rinse	500 ml amber glass	1	3	3
HCl rinse of permanganate impingers (if used)	500 ml amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	2
Nitric acid probe rinse (blank train)	500 ml amber glass	NA	NA	2
Acidified peroxide impinger solution and rinses (blank train)	500 ml amber glass	NA	NA	2
Initially empty impinger solution and rinse (blank train)	500 ml amber glass	NA	NA	2
Permanganate impinger solution and rinse (balnk train)	500 ml amber glass	NA	NA	2
HCl rinse of permanganate impingers (if used) (blank train)	500 ml amber glass	NA	NA	2
Nitric acid solution reagent blank	500 ml amber glass	NA	NA	1
Nitric acid/hydrogen peroxide solution reagent blank	500 ml amber glass	NA	NA	1
Acidified potassium permanganate solution reagent blank	500 ml amber glass	NA	NA	1
HCl solution reagent blank (if used)	500 ml amber glass	NA	NA	1
Filter blank	Glass petri dish	NA	NA	1
Subtotal		6		35
Stack Gas Hexavalent Chromium M0061				
Impinger solution and rinses	1 liter polyethylene	1	3	3
Filter and Residue	Glass vial	1	3	3
HNO3 Rinses	500 ml glass	1	3	3
KOH reagent blank	500 ml polyethylene	NA	NA	1
HNO3 Reagent Blank	500 ml polyethylene	NA	NA	1
Water reagent blank	500 ml polyethylene	NA	NA	1
Field spiked KOH solution (audit sample)	500 ml polyethylene	NA	NA	1
Subtotal		3		13
Stack gas Method 26A				
Filter	Petri dish	1	6	6
Front half acetone rinse	500 ml amber glass	1	6	6
Acid impinger liquid	500 ml amber glass	1	6	6
Alkaline impinger liquid	500 ml amber glass	1	6	6
Acetone reagent blank	500 ml amber glass	NA	NA	1
Sulfuric acid solution reagent blank	500 ml amber glass	NA	NA	1
Sodium hydroxide solution reagent blank	500 ml amber glass	NA	NA	1
Deionized water reagent blank	500 ml amber glass	NA	NA	1
Subtotal		4		28
Stack Gas M0010-TOE (TCO/GRAV)				
Filter	Glass petri dish	1	3	3
XAD-2 Resin trap	Glass trap	1	3	3
Front half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Back half acetone/methylene chloride rinses	500 ml amber glass	1	3	3
Condensate	1 liter amber glass	1	3	3
Filter (blank train)	Glass petri dish	NA	NA	1
XAD-2 Resin trap (blank train)	Glass trap	NA	NA	1
Front half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
Back half acetone/methylene chloride rinses (blank train)	500 ml amber glass	NA	NA	1
XAD-2 Resin trap blanks	Glass trap	NA	NA	2
Filter blank	Glass petri dish	NA	NA	1
acetone/methylene chloride reagent blank	500 ml amber glass	NA	NA	1
Subtotal		5		23

Table 6-2. Summary of Expected Trial Burn Field Samples

Sample Matrix	Container	Routine Samples or Field Splits (a) (No. per Run)	Number of Runs	Total Samples Collected During Test
Stack Gas M0040				
Sample bag	Tedlar bag	1	3	3
Condensate	40 ml VOA	1	3	3
Field blank bag	Tedlar bag	1	3	3
Condensate blank	Tedlar bag	1	3	3
Subtotal		4		12
Stack Gas PSD M5				
Filter	Perti dish	1	3	3
Subtotal		1		3
TOTAL		69		266

(a) "Field Splits" are separate portions of the same sample, placed into individual containers.
"Field Duplicates" are separate samples collected from the same sampling point.

Table 7-1. Sample Containers, Preservation, and Holding Times

Parameter	Sample Name	Containers	Preservation	Maximum Holding Time
Volatile organics	Makeup water, caustic, scrubber blowdown, POTW discharge	Glass VOA, Teflon-lined septum	Chill 4°C	14 days
	Spent Activated Carbon	Glass VOA, Teflon-lined septum	Chill 4°C	14 days
	Stack gas VOST tubes	Glass tube	Chill 4°C	14 days
	Stack gas VOST condensate	Glass VOA, Teflon-lined septum	Chill 4°C	14 days
	Stack gas M0040 bags	Tedlar bag	Protect from sunlight	72 hours
	Stack gas M0040 condensate	Glass VOA, Teflon- lined septum	Chill 4°C	14 days
SVOC, PAH, OCP, and PCB	Makeup water, caustic, scrubber blowdown (SVOC only), POTW discharge (SVOC only)	Glass bottle, Teflon- lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
	Spent Activated Carbon	Glass bottle, Teflon- lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0010 filter	Glass petri dish	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0010 sorbent tube	Standard cartridge wrapped in aluminum foil	Chill 4°C	14 days until extraction, 40 days after extraction
	Stack gas M0010 liquids	Glass bottle, Teflon-lined cap	Chill 4°C	14 days until extraction, 40 days after extraction
PCDD/PCDF	Stack gas M0023A filter	Glass petri dish	Chill 4°C	30 days until extraction, 45 days after extraction
	Stack gas M0023A sorbent tube	Standard cartridge wrapped in aluminum foil	Chill 4°C	30 days until extraction, 45 days after extraction
	Stack gas M0023A liquids	Glass bottle, Teflon-lined cap	Chill 4°C	30 days until extraction, 45 days after extraction
Metals (except Cr VI)	Makeup water, caustic, scrubber blowdown, POTW discharge	Glass bottle	NA	180 days/28 days for Hg
	Spent Activated Carbon	Glass bottle	NA	180 days/28 days for Hg
	Stack gas M29 filter	Petri dish	None required	180 days/28 days for Hg
	Stack gas M29 liquids	Glass bottle	None required	180 days/28 days for Hg
Hexavalent chromium	Stack gas M0061 liquids	Polyethylene bottle	Chill 4°C pH >8.5	14 days

Table 7-1. Sample Containers, Preservation, and Holding Times

Parameter	Sample Name	Containers	Preservation	Maximum Holding Time
Total Semivolatile and Non-volatile organics	Stack gas M0010 XAD-2 sorbant tube	Standard cartridge wrapped in aluminum foil	Chill 4^{o}C	14 days until extraction, 40 days after extraction
Total Semivolatile and Non-volatile organics Cont	Stack gas M0010	Glass petri dish	Chill 4^{o}C	14 days until extraction, 40 days after extraction
	Stack gas M0010 liquids	Glass bottle, Teflon-lined cap	Chill 4^{o}C	14 days until extraction, 40 days after extraction
Stack gas HCl/Cl_2	Stack gas M26A liquids	Glass bottle, Teflon lined cap	None required	30 days
Particle Size Distribution	Stack gas M5 filter	Petri dish	None required	NA
Stack gas particulate	Stack gas M26A	Petri dish	None required	NA
	Stack gas M26A front half rinses	Glass bottle, Teflon lined cap	None required	NA

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Spent Activated Carbon	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Chloride	1	3	SW846-5050	Ion chromatography (SW846-9056)
	Total metals	1	3	Acid digestion (SW846-3050)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
	Elemental	1	3	NA	(ASTM D5373) with (ASTM D3176) as an alternate
Makeup Water	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
Caustic	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
Scrubber Blowdown	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
POTW Discharge	Volatile Organics	1	3	Purge & Trap (SW846-5035)	GC/MS (SW846-8260)
	Semivolatile Organics	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	Total metals	1	3	Acid digestion (SW846-3020)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas M0030	VOCs + TICs (tenax + tenax/charcoal tubes) (Note 2)	(Note 3)	(Note 3)	Thermal desorption, trap (SW846-5041A)	GC/MS (SW846-8260)
	VOCs + TICs (condensate) (Note 2)	1	3	Purge and trap	GC/MS (SW846-8260)
Stack gas M0040	Total VOCs	1	3	Purge and trap for condensate Direct injection for gas	GC/FID (Guidance for Total Organics, App. A and E)
Stack gas M0010-SV (low res analysis)	Semivolatile Organics & TICs (Note 4)	1	3	Solvent extraction (SW846-3542)	GC/MS (SW846-8270)
	OCP (Note 5)	1	3	Solvent extraction (SW846-3542) & solvent exchanged to hexane or isooctane	GC (SW-846-8081)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M0010-P (high res analysis)	PCB (Note 7)	1	3	Solvent extraction (SW846-3542)	GC/MS (EPA Method 1668)
	PAH (Note 8)	1	3	Solvent extraction (CARB 429)	GC/MS (CARB 429)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas M0010-TOE	Total SVOCs	1	3	Solvent extraction (SW846-3542)	TOC GC/FID (Guidance for Total Organics, Appendix C)
	Total NVOCs	1	3	Solvent extraction (SW846-3542)	Gravimetric Method (Guidance for Total Organics, Appendix D)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M0023A	PCDD/PDCF	1	3	Solvent extraction (SW846-3500)	GC/MS (SW-846 Method 8290)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M29	Metals (Note 9)	1	3	Acid digestion (SW846-3050)	ICP (SW846-6020) & CVAAS (SW846-7470 for Hg)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M0061	Hexavalent chromium	1	3	NA	Ion chromatography, post-column reactor (SW846-7199)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Sample Name	Analysis	Samples per Run	Total Field Samples for Analysis	Preparation Method (See Note 1)	Analytical Method (See Note 1)
Stack gas M26A	Hydrogen chloride/Chlorine	1	3	NA	Ion chromatography (SW846-9057)
	Particulate	1	1	NA	Gravimetric (EPA Method 5)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Velocity	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas M00023A	PCDD/PCDF	1	3	Solvent extraction (SW846-8290)	GC/MS (SW846-8290; & SW846-0023A)
	Moisture	1	3	NA	Gravimetric (EPA Method 4)
	Temperature	1	3	NA	Thermocouple (EPA Method 2)
	Flow rate	NA	NA	NA	Pitot tube (EPA Method 2)
	Oxygen, Carbon dioxide	(Note 6)	(Note 6)	NA	Orsat or CEM (EPA Method 3)
Stack gas PSD	Particle size distribution	NA	NA	NA	Scanning Electron Microscope
Stack gas temporary CEMS	Total hydrocarbons	(Note 10)	(Note 10)	NA	Extractive Analyzers, EPA Method 25A
Stack gas Installed CEMs	Carbon Monoxide	(Note 10)	(Note 10)	NA	Extractive Analyzers, 40CFR 63 Appendix
	Oxygen	(Note 10)	(Note 10)	NA	Extractive Gas Analyzers, 40 CFR 63 Appendix

Note 1: "ASTM" refers to American Society for Testing and Materials, Annual Book of ASTM Standards, Annual Series.
"SW846" refers to Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and updates.
"EPA Methods" (Methods 1 through 5 and 23) refer to New Source Performance Standards, Test Methods and Procedures,, App. A, 40CFR 60.
"CARB" refers to California Air Resources Board methodology adopted January 27, 1987.
"Guidance for Total Organics" refers to EPA/600/R-96/036, March, 1996.

Note 2: Volatile Target Compounds as listed in this Test Plan, plus tentatively identified compounds.

Table 9-1. Summary of Performance Test Analytical Procedures and Methods

Note 3: During each sampling run, 4 pairs of VOST tubes (8 samples) will be collected, but only 3 pairs (6 samples) will be analyzed. The extra tube pair provides a contingency in case of breakage or other event that could require analysis of the extra tube pair. Analysis of each tube in each tube pair will be conducted separately.

Note 4: Semivolatile Target Compounds as listed in this Test Plan, plus tentatively identified compounds.

Note 5: Organochlorinated pesticide (OCP) target compounds as listed in this Test Plan.

Note 6: One set of gas bag samples collected during each stack traverse for Orsat analysis, or CEM.

Note 7: Polychlorinated Biphenyl (PCB) target compounds target compounds as listed in the Plan

Note:8 Polycyclic Aromatic Hydrocarbon (PAH) target compounds as listed in this Plan

Note 9: Metal Target Compounds as listed in this Test Plan.

Note 10: Installed CEMs sampling and analysis is continuous during each run.

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Total chloride	Spent Activated Carbon	3	Bomb or flask combustion (ASTM Method D-808 or E-442; SW-846 Method 5050)	Ion chromatography of residue (SW-846 Method 9056)	Duplicate	One per test	3	6
Ash	Spent Activated Carbon	3	NA	Residue after muffle furnace combustion (ASTM Method D-482)	Duplicate	One per test	1	4
Elemental	Spent Activated Carbon	3	Ultimate analysis (ASTM Method D-3176)	Ultimate analysis (ASTM Method D-3176)	Duplicate	One per test	1	4
Volatiles	Spent Activated Carbon	3	Solvent dispersion (SW-846 Method 5035/5030)	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl. Duplicates [c]	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	Scrubber blowdown	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl.	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	POTW discharge	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl.	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	Makeup Water	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl. Duplicates [c]	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	Caustic	3	NA	Purge and trap, GC/MS (SW-846 Method 8260)	Surrogate spikes	Every sample incl.	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	POHC Spike	3	NA	None; archive for analysis of required	NA	NA	NA	NA
	Analytical system QC	NA	NA	Purge and trap, GC/MS (SW-846 Method 8260)	LCS	One per batch/ matrix specific	2 or more	2 or more
					Method Blank	One per batch/ matrix specific	2 or more	2 or more

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatiles	Spent Activated Carbon	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates [c]	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	Scrubber blowdown	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates [c]	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	POTW discharge	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	Makeup Water	3	Waste Dilution(SW-846 Method 3580)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates [c]	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	Caustic	3	Liquid-Liquid Extraction (SW-846 Method 3510)	GC/MS (SW-846 Method 8270)	Surrogate spikes incl. duplicates [c]	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	Analytical system QC	NA		GC/MS (SW-8270)	LCS	One per batch/ matrix specific	2 or more	2 or more
					Method Blank	One per batch/ matrix specific	2 or more	2 or more
Metals by ICP	Spent Activated Carbon	3	Digestion (SW-846 Method 3051)	ICP (SW-846 Method 6010 or 6020)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	Scrubber blowdown	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	POTW discharge	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	Makeup Water	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Metals (Cont'd)	Caustic	3	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	Metals Spiking Solution	1	None; archive for analysis of required	None; archive for analysis of required	NA	NA	NA	NA
	Analytical system QC (aqueous)	NA	Digestion (SW-846 Method 3015)	ICP (SW-846 Method 6010 or 6020)	LCS	One per batch/ matrix specific	2 or more	6 or more
					Serial dillution	One per batch/ matrix specific	2 or more	
					Method Blank	One per batch/ matrix specific	2 or more	
	Analytical system QC (solids)	NA	Digestion (SW-846 Method 3051)	ICP (SW-846 Method 6010 or 6020)	LCS	One per batch/ matrix specific	2 or more	6 or more
					Serial dillution	One per batch/ matrix specific	2 or more	
					Method Blank	One per batch/ matrix specific	2 or more	
Mercury by CVAA	Spent Activated Carbon	3	CVAA (SW-846 Method 7471)	CVAA (SW-846 Method 7471)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	Makeup Water	3	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	Scrubber blowdown	3	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	POTW discharge	3	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	
	Caustic	3	CVAA (SW-846 Method 7471)	CVAA (SW-846 Method 7471)	MS [d]	One per test	1	6
					Duplicate or MSD	One per test	1	
					PDS [d]	One per test	1	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Mercury by CVAA (cont.)	Analytical system QC (aqueous)	NA	CVAA (SW-846 Method 7470)	CVAA (SW-846 Method 7471)	LCS	One per batch/ matrix specific	3 or more	3 or more
					Method Blank	One per batch/ matrix specific	2 or more	
	Analytical system QC (solids)	NA	CVAA (SW-846 Method 7471)	CVAA (SW-846 Method 7471)	LCS	One per batch/ matrix specific	3 or more	3 or more
					Method Blank	One per batch/ matrix specific	2 or more	
VOST for Volatile PICs and TICs	VOST stack sample tube pairs	9	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes [c]	Every sample	18	18
	VOST Condensate	3	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260)	Surrogate spikes [c]	Every sample	6	6
					Duplicate	One per test	1	
					MS/MSD [d,e]	One set test	2	
	VOST field blank tube pairs	1	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes [c]	Every sample	1	2
	VOST trip blank tube pairs	1	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes [c]	Every sample	1	2
	VOST audit sample tube pairs	2	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes [c]	Every sample	4	4
	Spiked resin blank tube pairs	2	NA	Purge and trap, GC/MS (SW-846 Methods 5041, 8260); Each tube in each tube pair is analyzed separately.	Surrogate spikes [c]	Every sample	2	4
	Analytical system QC	NA	NA	Purge and trap, GC/MS (SW-846 Method 8260)	LCS	1 per condensate batch	2 or more	2 or more
					Method blank	1 per analytical run	2 or more	2 or more

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Dioxin/Furans by Method 0023A	Method 0023A front half composite: filter, and front half of filter holder and probe rinses.	3	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	3	3
					Isotopically labeled surrogate spike	Every sample	3	
	Method 0023A back half composite: XAD-2 resin, and back half of filter holder and condenser rinses.	3	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	3	3
					Isotopically labeled surrogate spike	Every sample	3	
	Method 0023A blank train front half composite: filter, and front half of filter holder and probe rinses.	1	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
					Isotopically labeled surrogate spike	Every sample	1	
	Method 0023A blank train back half composite: XAD-2 resin, and back half of filter holder and condenser rinses.	1	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
					Isotopically labeled surrogate spike	Every sample	1	
	Method 0023A spiked XAD-2 resin blanks	2	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	2	2
					Isotopically labeled surrogate spike	Every sample	2	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Dioxin/Furans by Method 0023A (cont'd)	Method 0023A acetone reagent blanks	1	NA	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
	Method 0023A methylene chloride reagent blanks	1	NA	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
	Method 0023A toluene reagent blanks	1	NA	HRGC/HRMS (SW-846 Method 8290)	Isotope dilution internal standard spike	Every sample	1	1
	Analytical system QC	NA	Soxhlet extraction (SW-846 Method 0023A)	HRGC/HRMS (SW-846 Method 8290)	Method blank	1 per analytical batch	1 or more	1 or more
					Blank spike	2 per analytical batch	2	2
Metals by Method 29	Method 29 front half: filter, and nitric acid probe and front half filter holder rinses	3	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	PDS [d]	One per test	1	4
				CVAA (SW-846 Method 7471)	PDS [d]	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 10%HNO_3/5%H_2O_2 impinger contents and rinses	3	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	PDS [d]	One per test	1	4
				CVAA (SW-846 Method 7471)	PDS [d]	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 initially empty contents and rinses	3	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	PDS [d]	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 4%$KMnO_4$/10%H_2SO_4 impinger contents and rinses	3	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	PDS [d]	One per test	1	7
					Duplicate	Every sample	3	
	Method 29 4%$KMnO_4$/10%H_2SO_4 8N HCl rinses	3	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	PDS [d]	One per test	1	7
					Duplicate	Every sample	3	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Metals by Method 29 (cont'd)	Method 29 blank train front half: filter, and nitric acid probe and front half filter holder rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	MS [d]	One for test program	1	1
				CVAA (SW-846 Method 7471)	MS [d]	One for test program	1	1
				ICP (SW-846 Method 6010 or 6020)	MSD [d]	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD [d]	One for test program	1	1
	Method 29 blank train back half composite: 10%HNO_3/5%H_2O_2 impinger contents and rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	MS [d]	One for test program	1	1
				CVAA (SW-846 Method 7471)	MS [d]	One for test program	1	1
				ICP (SW-846 Method 6010 or 6020)	MSD [d]	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD [d]	One for test program	1	1
	Method 29 blank train initially empty contents and rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	MS [d]	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD [d]	One for test program	1	1
	Method 29 blank train 4%$KMnO_4$/10%H_2SO_4 impinger contents and rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	MS [d]	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD [d]	One for test program	1	1
	Method 29 blank train 4%$KMnO_4$/10%H_2SO_4 impinger 8N HCl rinses for MS/MSD analysis	2	Digestion (EMTIC TM-029)	CVAA (SW-846 Method 7471)	MS [d]	One for test program	1	1
				CVAA (SW-846 Method 7471)	MSD [d]	One for test program	1	1

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Metlas by Method 29 (cont'd)	Method 29 filter reagent blank	1	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	Reagent Blank	One for test program	1	1
				CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	Method 29 HNO_3 reagent blank	1	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	Reagent Blank	One for test program	1	1
				CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	Method 29 10%HNO_3/5%H_2O_2 reagent blank	1	Digestion (EMTIC TM-029)	ICP (SW-846 Method 6010 or 6020)	Reagent Blank	One for test program	1	1
				CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	4%$KMnO_4$/10%H_2SO_4 reagent blank	1	Digestion (SW-846 Method 3051)	CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	8N HCl reagent blank	1	Digestion (SW-846 Method 3051)	CVAA (SW-846 Method 7471)	Reagent Blank	One for test program	1	1
	Analytical system QC	NA		ICP (SW-846 Methods 3051, 6010 or 6020) and CVAA (SW-846 Method 7471)	LCS	one per batch/ matrix specific	1 or more	3 or more
					Serial dilution	one per batch/ matrix specific	1 or more	
					Method blank	one per batch/ matrix specific	1 or more	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (two separate trains collected and analyze. The first for PCB & PAH. The second for SVOC & OCP	Method 0010 front half composite: filter and probe rinses	3	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	3	3
					Semivolatile internal standard surrogate spikes	Every sample	3	
				GC for OCP (SW-846 Method 8081)	Semivolatile surrogate spikes	Every sample	3	3
					Semivolatile internal standard surrogate spikes	Every sample	3	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	3	3
					PAH recovery standard spike	Every sample	3	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	3	3
					PCB recovery standard spike	Every sample	3	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd) (two separate trains collected and analyze.) The first for PCB & PAH. The second for SVOC & OCP	Method 0010 back half composite: XAD-2 resin, condenser rinses	3	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	^{13}C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3
					Semivolatile surrogate spikes	Every sample	3	
					Semivolatile internal standard surrogate spikes	Every sample	3	
				GC for OCP (SW-846 Method 8081)	^{13}C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3
					organochlorinated pesticide surrogate spikes	Every sample	3	
					organochlorinated pesticide standard surrogate spikes	Every sample	3	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3
					PAH isotope dilution internal standard spike	Every sample	3	
					PAH recovery standard spike	Every sample	3	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	3	3
					PCB isotope dilution internal standard spike	Every sample	3	
					PCB recovery standard spike	Every sample	3	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Method 0010 condensate inpinger	3	Separatory funnel acid/base extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	3	3
					Semivolatile internal standard surrogate spikes	Every sample	3	
				GC for OCP (SW-846 Method 8081)	organochlorinated pesticide surrogate spikes	Every sample	3	3
					organochlorinated pesticide internal standard surrogate	Every sample	3	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	3	3
					PAH recovery standard spike	Every sample	3	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	3	3
					PCB recovery standard spike	Every sample	3	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Method 0010 blank train front half composite: filter and probe rinses	1	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	1	1
					Semivolatile internal standard surrogate spikes	Every sample	1	
				GC for OCP (SW-846 Method 8081)	organochlorinated surrogate spikes	Every sample	1	1
					organichlorinated pesticide internal standard surrogate spikes	Every sample	1	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	1	1
					PAH recovery standard spike	Every sample	1	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	1	1
					PCB recovery standard spike	Every sample	1	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Method 0010 blank train back half composite: XAD-2 resin, condenser rinses	1	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	^{13}C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	1	1
					Semivolatile surrogate spikes	Every sample	1	
					Semivolatile internal standard surrogate spikes	Every sample	1	
				GC for OCP (SW-846 Method 8081)	^{13}C-labeled pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	0	0
					Semivolatile surrogate spikes	Every sample	0	
					Semivolatile internal standard surrogate spikes	Every sample	0	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	1	1
					PAH isotope dilution internal standard spike	Every sample	1	
					PAH recovery standard spike	Every sample	1	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB pre-sampling surrogate spikes	Every XAD-2 resin tube before sampling	1	1
					PCB isotope dilution internal standard spike	Every sample	1	
					PCB recovery standard spike	Every sample	1	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Semivolatiles PICs and TICs, OCP, PAHs & PCBs by Method 0010 (cont'd)	Spiked resin blanks (Matrix spikes)	2	Soxhlet extraction (SW-846 Method 3542)	GC/MS for SVOCs (SW-846 Method 8270)	Semivolatile surrogate spikes	Every sample	2	2
					Semivolatile internal standard surrogate spikes	Every sample	2	
				GC for OCP (SW-846 Method 8081)	OCP surrogate spikes	Every sample	2	2
					OCP internal standard surrogate spikes (optional)	Every sample	2	
				HRGC/HRMS for PAHs (CARB Method 429)	PAH isotope dilution internal standard spike	Every sample	2	2
					PAH recovery standard spike	Every sample	2	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	PCB isotope dilution internal standard spike	Every sample	2	2
					PCB recovery standard spike	Every sample	2	
	Analytical system QC	NA	NA	GC/MS for SVOCs (SW-846 Method 8270)	LCS	1 per analytical batch	2 or more	4 or more
					Method Blank	1 per analytical batch	2 or more	
				GC got OCP (SW-846 Method 8081)	LCS	1 per analytical batch	2 or more	4 or more
					Method Blank	1 per analytical batch	2 or more	
				HRGC/HRMS for PAHs (CARB Method 429)	LCS	1 per analytical batch	2 or more	4 or more
					Method Blank	1 per analytical batch	2 or more	
				HRGC/HRMS for PCBs (EPA Draft Method 1668A)	LCS	1 per analytical batch	2 or more	4 or more
					Method Blank	1 per analytical batch	2 or more	

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Total semivolatile and nonvolatile organics by M0010 EPA TOE Guidance	Semivolatile front half composite: filter and solvent probe rinses	3	Soxhlet extraction (SW-846 Method 3542)	GC/FID analysis of one-half of pooled extracts (SW-846 Method 8015)	Duplicate	One per test	1	4
	Semivolatile back half composite: XAD-2 resin and condensor rinses	3		Gravimetric (Grav) analysis of one-half of pooled extracts (TOE Guidance)	Duplicate	Every sample	3	6
	Semivolatile condensate inpinger and rinses	3						
	Semivolatile blank train front half composite: filter and solvent probe rinses	1	Soxhlet extraction (SW-846 Method 3542)	GC/FID analysis of one-half of pooled extracts (SW-846 Method 8015)	Blank train	One per test	1	1
	Semivolatile blank train back half composite: XAD-2 resin and condensor rinses	1		Gravimetric (Grav) analysis of one-half of pooled extracts (TOE Guidance)		One per test analyzed in duplicate	2	2
	Methanol/ methylene chloride reagent blank	1	NA	GC/FID (SW-846 Method 8015)	Reagent blank	One per test	1	1
				Gravimetric (Grav) (TOE Guidance)		One per test analyzed in duplicate	2	2
	Filter blank	1	Soxhlet extraction (SW-846 Method 3542)	GC/FID (SW-846 Method 8015)	Reagent blank	One per test	1	1
				Gravimetric (Grav) (TOE Guidance)		One per test	1	1
	XAD-2 resin blanks	2	Soxhlet extraction (SW-846 Method 3542)	GC/FID (SW-846 Method 8015)	Reagent blank	One per test	2	2
				Gravimetric (Grav) (TOE Guidance)		One per test	2	2

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Total volatile organics by M0040	Tedlar Bags	6	NA	GC/FID (Modified SW-846 Method 0040)	Duplicate	Every sample	6	18
	Field BlankBag	3	NA	GC/FID (Modified SW-846 Method 0040)	Field blank	One per run	3	
	Condensate	3	NA	Purge and trap GC/FID (SW-846 Methods 0040 and 5030)	Duplicate	Every sample	3	8
	Condesate blank	1	NA	Purge and trap GC/FID (SW-846 Methods 0040 and 5030)	Field blank	One per run	1	
	Analytical system QC	NA	NA	GC/FID (Modified SW-846 Method 0040)	Zero gas	One per run	3	9
					Known gas	Two per run	6	
				Purge and trap GC/FID (SW-846 Methods 0040 and 5030)	Method blank (water)	One per analytical batch	1 or more	1 or more
HCl by Method 26A	Method 26A H_2SO_4 impingers	9		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	9	22
					MS/MSD analyzed in duplicate [d]	1 per batch (assuming all samples batched togeather)	4	
	Method 26A H_2SO_4 reagent blank	1		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	1	2
Cl_2 Method 26A	Method 26A NaOH impingers	9		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	9	22
					MS/MSD analyzed in duplicate [d]	1 per batch (assuming all samples batched togeather)	4	
	Method 26A NaOH reagent blank	1		Ion chromatography (SW-846 Method 9057)	Duplicate	Every sample	1	2

Table 10-1. Summary of Test Program Analyses

Analysis	Sample Matrix	Field Samples[a]	Preparation Method	Analytical Method	QC Analysis	QC Analysis Frequency [a]	QC Analyses	Total Analyses [b]
Cl^- ion chromotography	Analytical system QC			Ion chromatography (SW-846 Method 9057)	LCS/LCSD	1 per batch following initial callibration (separate calibration for each matrix)	4	8
					Method Blank	One per batch/ matrix specific - analyzed in duplicate	4	
Particle size distribution by Method 5 (SEM)	Filter	3		Scanning Electron Microscope	Blank Filter	Once per test	1	4
Particulate by Method 26A	Method 26A particulate filter	9		Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	9	9
	Method 26A probe and filter holder acetone	9		Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	9	9
	Method 26A acetone reagent blank	1		Gravimetric (Method 5)	Replicate weighing to constant weight	Every sample	1	1

[a] Each test condition is comprised of three replicate sampling runs. There is only one test condition planned for this test program. Refer to QAPP Tables 6-1 and 9-1.

[b] Total laboratory analyses includes field sample analyses and laboratory QC analyses

[c] Surrogate spikes are applied to all samples. Refer to Table 5-2 for the surrogate compounds.

[d] MS = Matrix spike
MS = Matrix spike duplicate
PDS = Post digestion spikes

[e] Refer to Table 5-2 for the matrix spike compounds.

Figure 11-1. Example Performance Test Report Outline

1.0 EXECUTIVE SUMMARY

2.0 TEST PROGRAM SUMMARY

2.1 Engineering Description

2.1.1 General Description
2.1.2 Residence Time Determination
2.1.3 Burner Description
2.1.4 Waste Feed Systems
2.1.5 Auxiliary Fuel System
2.1.6 Air Pollution Control System
2.1.7 Process Monitoring System (CMS)
2.1.8 Continuous Emissions Monitoring System (CEMS)
2.1.9 Automatic Waste Feed Cutoff System

2.2 Summary of Test Plan and Objectives
2.3 Test Implementation Summary

2.3.1 Deviations from the Test Plan

3.0 PROCESS OPERATIONS

3.1 Process Operating Conditions
3.2 Feed Material Characteristics
3.3 Feed Material Spiking
3.4 Effluent Characteristics

4.0 COMPLIANCE RESULTS

4.1 POHC Destruction and Removal Efficiency
4.2 Particulate Emissions
4.3 Hydrogen Chloride and Chlorine Emissions
4.4 Metals Emissions
4.5 Stack Gas Oxygen, Carbon Monoxide, and Total Hydrocarbons
4.6 Dioxin and Furan Emissions

5.0 QUALITY ASSURANCE/QUALITY CONTROL RESULTS

5.1 QA/QC Activities and Implementation

5.1.1 QA Surveillance
5.1.2 Sample Collection
5.1.3 Sample Analysis
5.1.4 Process Instrumentation
5.1.5 Stack Sampling Equipment
5.1.6 Laboratory Analytical Instrumentation

5.2 Audits and Data Validation
5.3 Calculations

Figure 11-1. Example Performance Test Report Outline

5.4 Conclusions

6.0 ANTICIPATED PERMIT OPERATING CONDITIONS

6.1 Development of Operating Limits
6.2 Specific Control Parameters

7.0 RECOMMENDED EMISSIONS DATA FOR USE IN RISK ASSESSMENT

7.1 Metals
7.2 Hydrogen Chloride and Chlorine
7.3 Particle Size Distribution
7.4 Speciated Volatile Organics
7.5 Total Volatile Organics
7.6 Speciated Semivolatile Organics
7.7 Total Semivolatile and Nonvolatile Organics
7.8 Dioxins and Furans
7.9 Speciated PAHs
7.10 Speciated Organochlorine Pesticides
7.11 Speciated PCBs

APPENDICES

A. Process Operating Data
B. Test Manager's Log
C. Spiking Report and Certificate of Analysis for Spiking Material
D. Process Instrument Calibration Data
E. Continuous Emissions Monitoring Data
F. Sampling Report
G. List of Samples
H. Analytical Report
I. Calculations
J. Documentation to Support Metals Extrapolation
K. Data Validation Report
L. Corrective Action Requests

Figure 14-2. Audit Checklist

Client:__________________ **Project No._________**

Date/Time of Audit: ________________________

System Audited:

____

Were applicable SOPs or methods followed? _______

Explain:
____
____
____
____

Was documentation where needed, complete and accurate? ______

Explain:
____
____
____
____

Were Chain of Custody procedures followed if applicable? _____

Explain:
____
____

Were applicable Health and Safety Requirements followed? _____

Explain:
____
____
____

Auditor's Signature/Date: ____